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EVALUATING THE STABILIZATION OF GYPSEOUS AND GYPSIFEROUS SANDS USING DIFFERENT CHEMICAL ADDITIVES TO MITIGATE GYPSUM DISSOLUTION

by

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Dedication

To my mother, for her love and support, my wife Aseel, for her encouragement, support, and love, and to my beloved son and daughters.

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Abstract

Gypsum, which is dihydrate calcium sulfate, ($CaSO_4.2H_2O$), is widely available in different sizes, from the size of a rock to the size of a few micrometers as a mineral, in different types of soils. It is primarily found mainly in arid and semi-arid areas around the world at different depths. Soils with gypsum and even gypsum rocks are very hard in their dry state. However, these soils and rock will experience remarkable dissolution upon wetting.

The dissolution phenomenon, which takes place in soils that contain gypsum, creates different geotechnical problems within the soil's profile, along with foundation issues for structures that have been constructed on this type of soil. Noticeable structural damage and collapses, which occurred in heavy and light structures, such as earthen dams, power plants, houses, and even roads, were related to the subsidence of soil with gypsum due to gypsum dissolution. The behavior that was exhibited by this problematic soil has captured the attention of civil engineers and scientists since the first quarter of the 20th century.

Many studies were conducted in different types of soils that contain gypsum within different soil profile depths to investigate the effects of this mineral on the properties of these soils. Those studies concluded that there are many factors that control the solubility of gypsum within the soil profile.

Studies that have been presented in this dissertation focused on the effects of the most important factors on the dissolution of gypsum, which are gypsum content, wetting-drying cycles, static and moving water. As a result, on soil stability, mass loss, porosity, and soil permeability were investigated.

Various tests and treatments were performed on two types of soils to study these effects. These soils are poorly graded sandy soils with different gypsum content that were brought from New Mexico. The first soil consisted of 93% gypsum (high gypsum soil), while the second soil consisted of 31% gypsum (medium gypsum soil).

First, these soils were classified, and then different types of hydration methods were used to measure the gypsum content. An evaluation of one of the dehydration method was conducted to select the best approach to measuring the gypsum content for different soil types, regardless of the size of the gypsum particles. Then, a measurement method was selected to determine the dissolved gypsum in the water and convert this measurement into a gypsum mass.

Three different additives (activated fly ash, asphalt emulsion, and Portland cement) were evaluated to select the appropriate additive that would enhance the mechanical properties of these soils. The selection of these additives was based on two criteria. First, additive must be inexpensive and available. Second, the impact of the additive on the environment and its ability to treat soil with gypsum was also considered.

Based on these criteria, two additives were chosen: fly ash and asphalt. Fly ash is a waste material that is produced after the combustion of coal in power plants.

Asphalt waste is a byproduct or sometimes is found in waste material that is produced from refining crude oil.

However, since asphalt binder requires high temperature in order to be melted and mixed with soil, and, considering safety concern and ease of use, a decision was made to use an anionic asphalt emulsion instead. A third option was to use Portland cement type I/II as a reference additive.

The studies were shown in four different objectives in Chapters 4, 5, 6, and 7. Chapter 4 discusses the activated fly ash treatment, in which 12 M of KOH was used to activate Class F fly ash to treat both soils. 10%, 20%, and 30% doses of activated fly ash were used in the treatment with curing periods of 7 and 28 days. Then, soil specimens went through 12 cycles of wetting and drying. The results showed that this treatment enhanced the soil stability and reduced the dissolution of gypsum. However, this treatment did not prevent or mitigate mass loss.

The second objective is discussed in Chapter 5. This chapter discusses the effects of static water on the dissolution of gypsum, and its results on soil stability, mass loss, and porosity for specimens that were treated with two different additives: anionic asphalt emulsion (6%, 12%, and 18%), and type I/II Portland Cement (9%). The results showed that the use of asphalt emulsions significantly improved soil stability, reduced gypsum dissolution and porosity, and mitigated mass loss when compared to the use of cement.

Chapter 6 discussed the effect of moving water on gypsum dissolution and soil permeability for both soils in three different states: soil in its natural condition, soil that was treated with 6% asphalt emulsion, and soil that was treated with 18% asphalt emulsion. Two different approaches were used to prepare and mix the asphalt emulsion with the soils. The first approach was conducted by mixing the soils with 6% asphalt emulsion, curing them, and then compacting them in constant head permeability cells.

The second approach was performed by mixing the soils with 18% asphalt emulsion, compacting them in PVC molds, and then curing them before placing them in flexible wall permeability cells.

The results showed that moving water had significant effect on the dissolution of gypsum, particularly for high gypsum soil. Moreover, the second treatment approach had a significant impact on soil permeability by reducing it to a state where water no longer flowed through the high gypsum soil. This approach also led to a reduction in gypsum dissolution for both soils.

The unconfined compressive strength for different specimens treated with activated fly ash, asphalt emulsion, and Portland cement were measured and compared with each other, as described in Chapter 7. Although the results suggested that the highest strength was achieved by treating both soils with 9% Portland cement, this option is not preferable due the sulfate attack that occurred in the soil upon wetting due to the presence of gypsum. The use of asphalt emulsion remained the best and preferred treatment.

Finally, Chapter 8 provides the conclusions and the recommendations, based on the results that were provided. that the results of these studies suggested. The outcome of the studies that have been presented in this dissertation show that gypsum content has a significant impact on soil stability.

Moreover, moving water contributes to the deterioration of these soils by increasing the dissolution of gypsum, which suggest that and preventing direct contact between the water and the gypsum particles will enhance soil stability. The effects of wetting-drying cycles can be mitigated by using asphalt emulsions or asphalt binders. The use of asphalt emulsions enhances different properties for soil with gypsum.

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Chapter 1

Introduction

Soil with gypsum has been found in many countries in different continents: Africa, southern and central Asia, Europe, and North America (FAO 1990). Figure 1.1 shows the global distribution of soil with gypsum. In the U.S., soils with gypsum have been identified in the western part of the continent, which has been classified as an arid and semi-arid region (Pearson et al. 2015). In Iraq, which has a low rate of precipitation, soils with gypsum represent the highest percentage of soils in all Iraqi regions (Buringh 1960; Porta 1998; Taha et al. 2008; Al-Layla and Al-Saffar 2014).

The engineering behavior of gypsum soils is highly related to seasonal changes, specifically the wetting and drying cycles (Buringh 1960; Porta 1998; Fattah et al. 2008; Fattah et al. 2012; Awn 2011; Khattab and Hussein 2012; Alateya 2013; Razouki and Salem 2014; Salih, 2013).

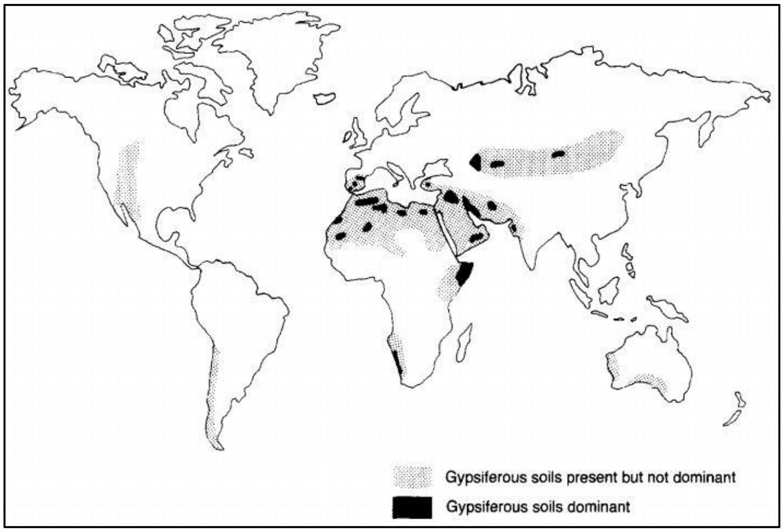


Figure 1.1 Global distribution of soil with gypsum (Boyadgiev and Verheye, 1996).

1.1 Problem statement

Soils containing gypsum have attracted the attention of civil engineers due to the damage that can be caused to infrastructures supported on these soils, including light weight structures (e.g., roads) and heavy structures (e.g., dams) (Porta 1998; Taha et al., 2008; Fattah et al., 2008; Fattah et al., 2013). For example, the presence of gypsum karst led to the development of cavities and subsequent collapses under roads and bridges In Northern England (Cooper and Saunders 2002).

The primary issues with these soils are high solubility, compressibility, and collapsibility. The solubility (dissolution) of gypsum within the soil, due to water flow, can lead to increases in pore size and volume. This phenomenon, coupled with the applied structural loads, increases the probability of cavity creation that can accelerate soil compressibility collapsibility (Cooper and Saunders 2002; Poch et al., 1998). Soils with a higher coefficient of permeability, such as sands, are particularly susceptible to gypsum dissolution.

Gypsum soils can be stabilized with chemical additives to control dissolution. In this dissertation, the effects of different additives on sands with medium to high gypsum contents are investigated. The additives that were selected were asphalt emulsion, activated fly ash, and Portland cement, which was used as a control additive.

1.2 Research questions

This study is intended to answer the following research questions:

1- How can gypsum sands be stabilized with chemical additives, such as asphalt emulsion and activated fly ash, to control volume changes within the range of ±2.5% and mass losses of less than 7% when exposed to repeated wetting and drying?

2- How do the amounts and types of chemical additives, such as asphalt emulsion and activated fly ash, affect the amount of gypsum that dissolves in gypsum sands exposed to repeated wetting and drying?

3- How does repeated wetting and drying of gypsum sands treated with increasing amounts of asphalt emulsion change the permeable porosity?

4- What are the effects of gypsum content and gypsum dissolution on the coefficient of permeability of untreated and treated gypsum sands exposed to continuous water flow, and how well can changes in the coefficient of permeability be predicted?

5- How will continuous flow affect the amount and rate of gypsum dissolution for medium and high gypsum sands that are treated with asphalt emulsion?

6- What is the effect of repeated wetting and drying on the relationship between gypsum content and unconfined compressive strength of gypsum sands treated with asphalt emulsion and activated fly ash?

1.3 Dissertation structure

This research has been divided into four different objectives. Each objective is addressed in a separate chapter.

Chapters 2 and 3 discuss prior work that has been done to classify gypsum soils and estimate gypsum content, along with all the materials and equipment that have been used to support this research.

In Chapter 4, which covers the first objective, the use of Class F activated fly ash as an additive to stabilize two gypsum sands was studied. This study included volume stability, mass loss, and gypsum dissolution through wetting-drying cycles, and the effect of these cycles on the unconfined compressive strength of treated specimens.

The second objective is discussed in Chapter 5. This objective investigates the effect of static water during wetting-drying cycles on the dissolution of gypsum for soils specimens were treated with asphalt emulsion as a stabilizer. Volume changes, water content, permeable porosity, and mass loss are also presented in this chapter.

The effect of continuous water flow was evaluated on untreated specimens and specimens stabilized with asphalt emulsion. The constant head permeability test was used to measure the coefficient of permeability, and the leachate was collected to measure the amount of dissolved gypsum with time. These results are presented in Chapter 6. As part of the third objective.

Chapter 7 covers the fourth objective, which compares the unconfined compressive strength for specimens treated with the three different additives: asphalt emulsion, activated fly ash, and Portland cement.

Chapter 8 provides the conclusions and recommendations for future research, based on the findings of this study.

Chapter 2

Background

2.1 Gypsum in soil

Soils with gypsum as a component are found in arid and semi-aired regions around the world. Gypsum is found in these soils in the form of hydrated calcium sulfate (CaSO4.2H2O) (Buringh 1960; Cooper and Saunders 2002; Herrero et al. 2009; Porta 1998). Gypseous (Gypsiferous, Gypsosols, Gypsic) soil was discovered by German soil scientist, W. Knop in 1871. At that time, he classified it as a sulfate soil, (genus gypsic soils) (Boyadgiev and Verheye 1996).

Gypsum, or dihydrate calcium sulfate, ($CaSO_4.2H_2O$), is one of the five solid phases for the calcium sulfate system $CaSO_4$-H_2O. The second phase is basanite (plaster of Paris), which is known as hemihydrate calcium sulfate ($CaSO_4.0.5H_2O$). The other phases are anhydrate I (α-$CaSO_4$), anhydrate II (natural anhydrate β-$CaSO_4$), and anhydrate III (soluble anhydrate γ-$CaSO_4$).

Gypsum is the most available phase of calcium sulfate in soil, compared to hemihydrate, which is very rare in soil because it will transform to gypsum in the presence of water or humidity at atmospheric pressure. The anhydrate was found in deep deposits with gypsum, in the cap rocks of salt domes, and in ancient marine evaporate deposits (Casby-Horton et al. 2015).

The arrangement of the $CaSO_4$-H_2O crystalline structure consists of chains of alternating calcium atoms and sulfates tetrahedra coordinated through oxygen atoms, as listed by Charola et.al. 2007 and cited by Casby-Horton et al. 2015. Both gypsum and hemihydrate have a sheet of water molecules coordinated between chains of calcium and sulfates, as shown in (Figure 2.1).

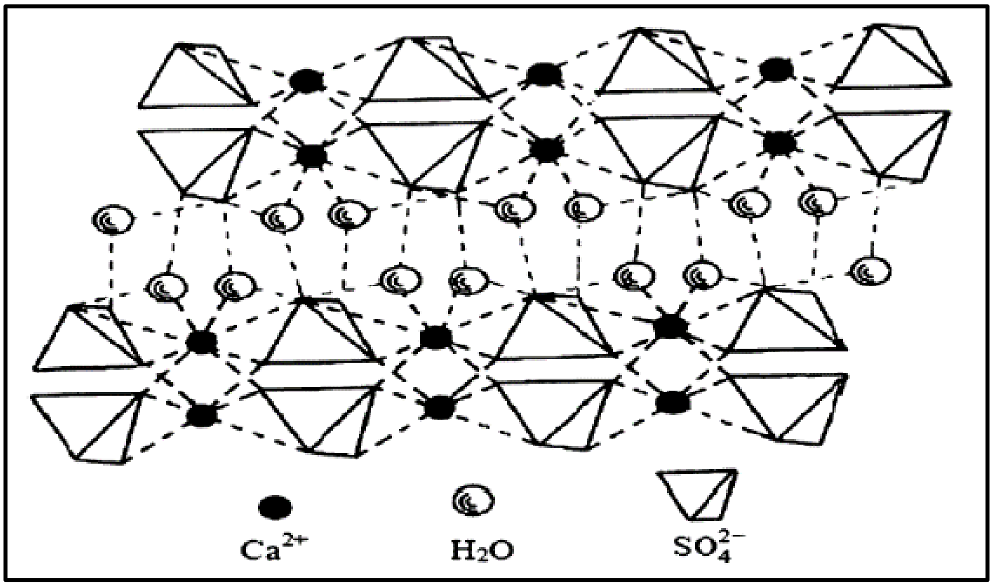


Figure 2.1 Gypsum crystal structure (Chen 2006 and cited by Yu et al. 2015).

While in the anhydrate, the crystal is most closely packed, as well as most dense, provides the most stability and the lowest reactivity between all the $CaSO_4$-H_2O phases (Casby-Horton et al. 2015).

Gypsum, when it is dry from free moisture, contains approximately 21% chemically bound water in its crystal matrix. To evaporate the free moisture, gypsum samples must be heated at 40ºC for 24 hrs. (Thomas 2002).

2.2 Classification of gypsum in soil

Buringh (1960) classified gypsum in soil as two types: primary gypsum and secondary gypsum.

Primary gypsum represents the gypsum rocks that typically form the bed-rock of the soil with gypsum. Secondary gypsum represents the other forms of gypsum inside the soil. There are two types of secondary gypsum: the crystalline and the amorphous gypsum.

In many cases, the crystalline gypsum appears as a formation called selenite, which are colorless and transparent monoclinic crystals. Figure 2.2 shows soil with gypsum components from two different regions in the world.

a. Gypseous soil layers with gypsum rock from Sulaimani /Iraq.

b. Gypseous soil from Aust Cliff Gypsum (UK).

c. Gypsum rock from Aust Cliff Gypsum (UK).

Figure 2.2 Gypseous soils components from two different regions in the world (Salih 2013).

The size of gypsum in the soil covers a large range, from the size of the parent rocks to the size of gypsum crystals. Therefore, the gypsum inside the soil profile can be recognized by the naked eye to scanning by the electronic microscope.

The gypsum crystals were classified into three groups, based on size: spar (> 20 µm), microspar (5-20 µm), and micrite (< 5 µm) (Jafarzadeh and Burnham 1992).

In general, the presence of gypsum in soil comes from gypsum deposits, which formed geologically, and secondary gypsum, which migrated and accumulated inside the soil profile.

2.2.1 Factors controlled gypsum migration inside soil profile

Secondary gypsum migration in the soil is controlled by the following factors:

a- The irrigation of crops.

b- The movement of the water inside the soil profile.

c- Variation of the water table during seasonal changes.

d- Movement of the surface water.

These factors will increase the weathering rate of the parent gypsum rock, thus relocating gypsum inside the soil profile and close to the ground surface (FAO 1990; Salih 2013).

Gypsum content in these soils varies from less than 1% to almost 100%. Therefore, they have been classified, per their gypsum content, as gypsiferous soil when the gypsum content < 40%, and gypseous soils when the gypsum content > 40% (Pearson et al. 2015). To identify these types of soils, the gypsum content must be calculated first.

2.3 Classification methods for soil with gypsum

There are different methods that are used to classify the soils, and each method consists of two parts. The first part is the sieve analysis, which is used to classify the soil particle sizes that range from gravel to sand (e.g. 75 to 0.075 mm). The next part is the hydrometer analysis, which is used to classify soil particles that are smaller than sand (< 0.075mm).

The most difficult soil particles to classify in soil with gypsum are those whose sizes range between sand and clay sized particles. In general, gypsum particle sizes fall in this range. Therefore, the part of the soil classification process that needs to be modified is the hydrometer analysis.

In the published literature, different methods were used to classify gypseous soils. Some of these methods used ordinary classification methods to classify these soils. These methods were supported by saying that gypsum between the soil particles is considered to be part of the soil structure, and if it is removed or modified during testing, the soil will lose one of its components. Fattah et al. (2008) said that gypsum has a big impact on the physical properties of the soil. Therefore, using any method to remove it or to prevent it from dissolving during classification will affect these properties.

Other researchers preferred to dissolve and remove the gypsum from the soil before classification. They argued that the presence of gypsum will result in a soil classification that does not represent the true grain size distribution.

AlNouri and Saleam (1994) used the Ethylenedinitrilotetraacetic acid (EDTA) solution method (Bodine and Fernalld, 1973 method) to remove the gypsum from the soil to prevent flocculation during the hydrometer test.

The last group used specific methods to classify soil without gypsum dissolution. Using water during the hydrometer test will dissolve the gypsum, which will result in a classification that does not represent the soil. Therefore, other coating chemicals or other liquids have been used during the hydrometer test.

Razouki et al. (2012) evaluated the problem of flocculation during the hydrometer test due to the presence of calcium ions, which come from gypsum.

For this reason, they used barium sulfate ($BaSO_4$) and barium chloride ($BaCl_2$) (Hesse, 1974 method, listed in Razouki et al., 2012) to coat the gypsum particles prior to the hydrometer test.

2.3.1 Standard soil classification systems

In general, there are many systems that can be used to classify soils. The most common system is the Unified Soil Classification System (USCS, ASTM D2487-10). This standard is used to classify all the soils and rocks according to particle size distribution, liquid limits, and plastic limits.

However, many ASTM standards also have been used to classify soils, such as ASTM D422-63. British standards are also used to classify different types of soils and rocks. These standards include (BS 1377:1990 and BS 5930:1999). The American Association of State Highway and Transportation Officials (AASHTO) also has a system that is used to classify soils that re related to roadways construction.

2.3.2 Classifying soil after dissolving and removing the gypsum

Various methods have been used to remove gypsum from the soil. For example, the EDTA method, (Bodine and Fernald, 1973), is performed by mixing the soil with a solvent produced by boiling ethylenedinitrilotetraacetic acid (EDTA) at a pH of 10-12. The Dilute hydrochloric acid (HCl) method (Loveday, 1974 listed in; Bashour and Sayegh, 2007) has also been used to remove the gypsum from soil.

This method is used on soils with visible gypsum crystals. This method is performed by adding 25 ml of 2M hydrochloric acid (HCl) to 25 g air-dried soil in a flask.

The process also requires the addition of 5 drops of hydrogen peroxide (H_2O_2), 500 ml of distilled water, 5 ml of 1M sodium hydroxide (NaOH), and 10% sodium-hexametaphosphate. After this process is completed, all the gypsum will be removed from the solution (Bashour and Sayegh 2007).

2.3.3 Classification after coating the gypsum to prevent dissolution / flocculation of gypsum particles prior to standard testing

The barium sulfate ($BaSO_4$) and barium chloride ($BaCl_2$) method, (Hesse, 1974) uses soil samples that are treated with barium chloride ($BaCl_2$), followed by barium sulfate ($BaSO_4$) as a coating, to prevent the dissolution of gypsum particles before testing (Razouki et al., 2012).

The Kerosene method was used by Fattah et al. (2008) which relies on the procedure outlined by Bowles (1978). The USCS system is used to classify soils, but kerosene is used instead of water during the grain size distribution analysis to prevent gypsum dissolution.

The last method is the ethanol: water solution method, which is explained by Pearson et al. (2015). This method uses a solution of 7:3 ethanol-to-water ratio during its grain size distribution analysis.

Table 2.1 provides various factors that must be taken into consideration when choosing the best classification method. From this table, gypsum removing methods are better suited, because the test time is reasonable and not as complex as the gypsum coating methods. Moreover, these methods have a lower cost than the coating methods.

Table 2.1 Soil classification methods evaluation

Method	Complexity	Cost	Test time	Accuracy	Chemicals used
Ordinary	Low	Low	Short	Low	Non
Gypsum removing	Medium	Medium	Medium	Medium	High
Gypsum coating	High	High	Long	Medium	High

2.4 Gypsum content of soil

Many methods have been used to calculate the gypsum content. In general, they can be classified as chemical and dehydration methods.

There are many types of chemical methods that have been used to calculate the gypsum content in the soil. The standard SO_4 method was developed by the Soil Conservation Service in 1972 (method 6L1b, and the corrected method 6F1b, 1972). In this method, the gypsum content is calculated by measuring the total SO_4 in the water/soil solution after dissolving all the gypsum in the soil (Nelson et al. 1978).

The British standard method calculates the gypsum from the sulfate content by multiplying this content with a constant factor (BS 1377:1990 part 3, 1990). Another method, called the acetone method, uses acetone to precipitate the gypsum from the filtered water/soil solution.

This method requires a variety of chemicals and several sensitive devices (NRCS, USDA report No.42, 2014). Concisely, the chemical methods are complicated and require sensitive devices, along with different types of chemicals.

On the other hand, the dehydration methods are simple and do not require highly sensitive equipment. These methods performed by heating the soil to a temperature that ensures the evaporation of the two water molecules in the gypsum crystal.

By calculating the percentage change in weight before and after heating, the gypsum content can be determined. Gypsum dehydration has been investigated extensively during the last two decades due to the use of gypsum as one of the components in gypsum board and wood or steel studs, which are used as walls, floors, and ceilings in different types of buildings. Although these materials are used to isolate different spaces, they also work as fire resistance.

The gypsum dehydration process will delay or slow down fire, which provides more time for evacuation. The additional time for evacuation occurs because the transfer of heat inside these materials is delayed until the gypsum dehydration process is completed (Kontogeorgos et al., 2011; Kolaitis and Founti 2013).

Gypsum, when it is dry from free moisture, has approximately 21% chemically bound water in the crystal matrix. To evaporate the free moisture, gypsum samples must be heated in 40ºC for 24 hrs. (Thomas 2002). Gypsum dehydration or calcination occurs at temperatures ranging from 80ºC to 250ºC (Kolaitis and Founti 2013). Depending on the vapor pressure, which has a significant effect on this process, gypsum during dehydration will transfer into other $CaSO_4$-H_2O phases.

When the vapor pressure is close to zero, the water vapor can escape freely, and the gypsum will start to dehydrate. van der Heijden et al. (2011) showed that the chemically bound gypsum water starts to evaporate at 100ºC, and the anhydrate will be formed.

$$CaSO_4.2H_2O(s) + \Delta H_{dehyd} \rightarrow CaSO_4(s) + 2H_2O(g) \qquad \text{(Eq. 2.1)}$$

This reaction needs a total energy of 625 kJ/kg of dry gypsum. 150 kJ/kg is needed to separate the water molecules from gypsum crystal structure, and the remaining energy (475 kJ/kg) is needed to evaporate the released water. This reaction will occur between 90ºC to 150ºC. When the water vapor is not allowed to escape freely (i.e. a closed system), then two endothermic decomposition reactions will occur during gypsum dehydration (Kolaitis and Founti 2013; van der Heijden et al., 2011).

Reaction One:

$$CaSO_4.2H_2O + \Delta H_{dehyd,1} \rightarrow CaSO_4.\frac{1}{2}H_2O + \frac{3}{2}H_2O \qquad \text{(Eq. 2.2)}$$

Reaction Two:

$$CaSO_4.\frac{1}{2}H_2O + \Delta H_{dehyd,2} \rightarrow CaSO_4 + \frac{1}{2}H_2O \qquad \text{(Eq. 2.3)}$$

During the first step (Reaction One), gypsum is partially dehydrated and loses 75% of the chemically bound water to form the hemihydrate, and the energy used during this step is approximately 450 kJ/kg. In the second step (Reaction Two), the hemihydrate dehydrates and loses its remaining water to form type III anhydrate.

The temperatures that lead to these reactions are highly dependent on the vapor pressure (van der Heijden et al. 2011; Kolaitis and Founti 2013). van der Heijden et al. (2011) showed that both reactions occurred in a range of temperatures between 125ºC and 225ºC. Meanwhile, Kolaitis and Founti (2013) showed that Reaction One occurred at a temperature of 156ºC, and Reaction Two occurred at a temperature of 192ºC. At a temperature of 400ºC, the molecular structure of the soluble crystal (anhydrite III) will change and undergo additional decomposition reactions (Kontogeorgos and Founti 2012). Therefore, any dehydration process that is used to find the gypsum content must be below this temperature.

Other factors that affect gypsum dehydration are time and gypsum grain size. Khalil (1982) found that gypsum dehydration increases with an increase in time and temperature, along with a reduction in the gypsum grain size. Moreover, for a constant gypsum size, the dehydration reaction at 160ºC is double of the 100ºC and four times the reaction at 70ºC.

From the author's results, it can be inferred that, at a temperature ≥ 140ºC and heating duration ≥ 5 hrs., the gypsum content calculated for different pure gypsum grain sizes samples was ≥ 98 %.

In his study, Khalil used five dehydration temperatures (100, 120, 140, 160, and 180 ºC), to calculate the dehydrated gypsum fraction (α) for seven gypsum samples with sizes of 0.088, 0.0965, 0.127, 0.222, 0.649, 1.204, and 2.109 mm for dehydration times of 0.25, 0.5, 1, 2, 5, and 10 hrs. The results showed that the gypsum particles need to be as fine as possible to calculate the gypsum content using the shortest period.

Moreover, from Table 2.2 , which shows the results for 0.25, 0.5, and 1 hr. dehydration times for the particle sizes of 0.088 and 2.109 mm, a dehydration time of 0.25 hr. (15 min.) was not enough to dehydrate the gypsum, even for the finest gypsum size, at the highest dehydration temperature. The dehydrated gypsum for the gypsum particle with a size of 0.088 mm and a dehydration temperature of 180º C for 0.25 hr. dehydration time was 98.87 %).

However, for the same size, the dehydration time of 0.5 hr. (30 min.) was acceptable because the dehydrated gypsum at a temperature of 180ºC was 99.07%. However, it did not effectively estimate the dehydrated gypsum for sizes larger than 0.222 mm.

Table 2.2 Results from Khalil (1982)

Gypsum particle size of 0.088 mm.

Temp. (ºC)	**Time (hr.) / α**		
	0.25	**0.5**	**1**
100	2.5	7.83	40.46
120	23.58	51.66	90.12
140	62.67	80.04	97.85
160	84.39	99.12	99.12
180	98.87	99.07	99.22

Gypsum particle size of 2.109 mm

Temp. (ºC)	**Time (hr.) / α**		
	0.25	**0.5**	**1**
100	0.59	1.08	7.79
120	11.84	37.18	67.17
140	38.16	69.47	93.44
160	48.09	88.16	94.18
180	90.07	96.72	98.83

Therefore, the results provided by Khalil (1982) suggested that for all the seven gypsum particle sizes, a dehydrated temperature of 180 ºC and a dehydration time of 1 hour or more in a well-ventilated oven (zero vapor pressure) will ensure the dehydration of all the gypsum particles.

As a result, from all these studies, calculating gypsum content using dehydration method should be done with zero vapor pressure to ensure that all the gypsum will transfer directly to the anhydrite III (Eq. 1). If any vapor pressure is initiated during the dehydration, the two reactions (Eq. 2 and 3) will occur, which will give misleading gypsum content results.

The temperature and the duration of heating vary for different dehydration methods. Nelson et al. (1978) method used 105º C and a heating duration of 24 hrs. Al-Mufty and Nashat (2000) method (listed in Fattah et al. 2012), used 110º C, with the duration depending on when the weight reached a constant value after heating. The OMRAN (2016) method heated the soil at 150º C for 15 min. Table 2.3 shows the details of these three methods.

Table 2.3 The dehydration methods

No	Dehydration method	Soil drying			Soil heating		
		Temp	Time	Device	Temp.	Time	Device
1	Nelson et al. (1978)	Room	48 hrs.	desiccator	105º	24 hrs.	Oven
2	Al-Mufty & Nashat (2000)	45ºC	Constant Weight	Oven	110ºC	Constant Weight	Oven
3	Omran (2016)	70ºC	45 min.	Oven	150ºC	15 min.	Oven

2.5 Problems related to gypsum in soil

2.5.1 Gypsum dissolution

Gypsum dissolution is related to its solubility in water. The gypsum solubility rate at 25°C and a pressure of 0.101 MPa in pure water is approximately 2.6 g/L (Eswaran and Zi-Tong 1991). When gypsum is in contact with water, it will dissolve into calcium ions and sulfate ions (Fattah et al. 2008). However, the solubility of gypsum in the soil is controlled by many factors as follows:

2.5.1.1 Factors affect on gypsum solubility in soil with gypsum

a- Particle size: Khan (1994) found that the solubility of gypsum in soils is dependent on the particle size. He concluded that the solubility would increase with a reduction in the particle size. He attributed this reduction to the increase in the total surface area with the reduction of the particle size, which will result in an increase in gypsum solubility in water.

Porta (1998) concluded that gypsum solubility will be low when the gypsum crystals are relatively large. Sonnenfeld (1984), cited by Salih (2013), reported that maximum gypsum solubility will occur when the gypsum crystals size between 0.2-0.5 μm.

- **b- Dilution ratio:** Khan (1994) also found that the solubility of gypsum increases with increase in the soil: water ratio because more gypsum will dissolve due to this increase. Van Alphen and Romero (1971), cited by Kuttah and Sato (2015), found that a dilution ratio of 1:1 will only dissolve 0.25 % of the gypsum. Therefore, this ratio must be very high to dissolve all the gypsum in the soil.

  They also reported that this ratio is related to the gypsum content, and they gave an example of soil with 40% gypsum content, for this soil and to dissolve all the gypsum, the dilution ratio must be greater than 1:160.

- **c- Temperature:** Casby-Horton et al. (2015) reported that at a temperature greater than 42° C, gypsum will be less soluble than at a temperature less than 42° C, as shown in Figure 2.3. James and Lupton (1978), cited by Kuttah and Sato (2015), concluded that the gypsum solubility rate increases by 3.25 times when the temperature is increased from 5° C to 23° C.

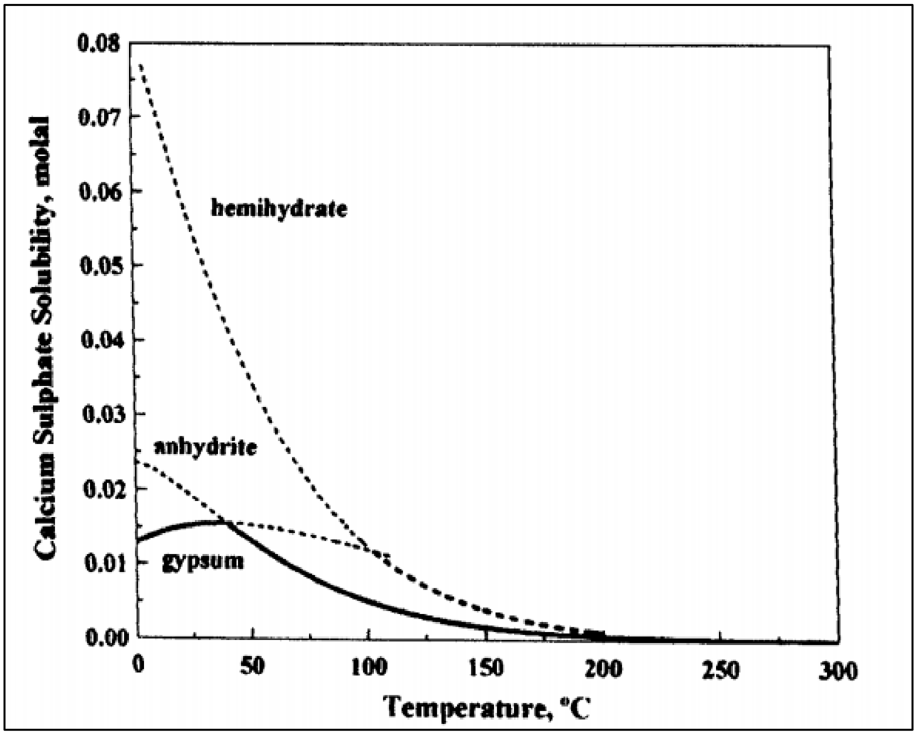


Figure 2.3 The solubility of gypsum, hemihydrate, and anhydrite with temperature (Azimi et al. 2007 cited by Casby-Horton et al. 2015).

d- Other salts in soil: In pure water, the solubility of gypsum is approximately 2.6 kg/m^3 (i.e.: 2.6 g/l at 25° C and a pressure of 0.101 MPa), but the presence of other salts in soil will change this value (Doner and Lynn 1989; and McFadden et al. 1991, cited by Boyadgiev and Verheye 1996).

The USDA Soil Survey Investigations Report No. 42, (2014) shows that the solubility of NaCl salt in water at 20° C is 360 g/l, which is very high compared to the solubility of gypsum. Buringh (1960) said that the solubility of gypsum in pure water is relatively low compared to the solubility of other salts in the soil. Therefore, in many cases, the water inside the soil is considered to be saline due to the high concentration of salts other than gypsum.

He classified magnesium chloride, sodium chloride, calcium chloride, magnesium sulfate and sodium sulfate as very high soluble salts, and he classified calcium sulfate, magnesium carbonate, and calcium carbonate as low soluble salts in soils. However, he also explained that the water in the soil has a different mixture of salts with different concentrations because each type is related to the availability and the concentration of other salts in the soil.

Barzanji (1973), cited by Fattah et al. (2008) listed that the solubility of gypsum in the soil will increase if the water has a concentration of sodium chloride and magnesium chloride, but it will be decreased if calcium bicarbonate is in the soil. Al-Neami (2006) and Nasir (2008), cited by Karim et al. (2012), stated that the solubility of gypsum is controlled by the chemical properties of the seepage water in the soil.

Hardie (1967) and López et al. (1999), cited by Morillas et al. (2009), said that after submerging gypseous soil samples in NaCl solution, the solubility of gypsum was three times the normal value.

Elrashidi et al. (2007) stated that due to the high solubility of other salts in soils, they need to be considered with gypsum when evaluating the subsidence problem of soils. The USDA Soil Survey Investigations Report No. 42 (2014) lists that the solubility of gypsum in saline water is approximately 20-50 meq/l (milliequivalents/liter). Lagerwerff et al. (1965) cited by Porta (1998), stated that in instances where Ca^{+2} or $SO4^{-2}$ ions are available in the soil from a source other than gypsum, the solubility of gypsum will be reduced.

e- **Flow Rate:** Kemper et al. (1975), cited by Fattah et al. (2008), showed that the solubility of gypsum is related to the flow rate of water inside the soil.

f- **Soil Permeability:** Al-Neami (2006) and Nasir (2008), cited by Karim et al. (2012), stated that soil permeability will affect gypsum solubility because it will control the amount of water inside the soil, along with its movement.

The relation between soil permeability and gypsum solubility is complicated. Kuttah and Sato (2015) mentioned that soil permeability will increase with the increase of gypsum content only when the gypsum particles are larger than the soil particles because the solubility of gypsum particles will increase the flow of water in the soil. On the other hand, soil permeability will decrease with an increase in gypsum content when the gypsum particles are smaller than the soil particles because the gypsum particles will close the paths of water in the soil.

g- **Applied pressure:** Freyer and Voigt (2003) pointed out that the solubility of all $CaSO_4$ salts will increase with an increase in the applied pressure.

h- **pH of soil:** The influence of the pH on gypsum solubility is not clear. Subhi, (1987), cited by Kuttah and Sato (2015), found that acidity will increase the solubility of many salts in the soil. Shlash and Al-Rawi (1994), cited by Kuttah and Sato (2015), reported that treating gypsiferous soils with different concentrations of nitric acid (HNO_3) and hydrochloric acid (HCl) will reduce the presence of many of the salts in the soil, such as $CaSO_4$.

2.5.2 Soil compressibility

"Soil compressibility is the capacity of soil to decrease in volume when subjected to a mechanical load. The process that describes the decrease in soil volume (soil densification) under an external applied load is called compression. An externally applied load can be in the form of a static load or a dynamic load." (Gupta et al. 2002)

Compression occurs in soil due to:

a- Air and water exclusion from void space.

b- Soil particles rearrangement.

c- Compression and deformation solid particles and soil aggregate.

d- Liquid and gas compression inside the soil voids. (Gupta et al., 2002)

In instances where gypsum is present in the soil and with the application of external loads, the soil will experience compression due to the crushing and rearrangement of gypsum particles, particularly larger particles (Salih 2013).

The factor that has been used in many studies to describe soil compressibility is the compression index (c_c), due to its direct relationship to consolidation settlement in soils with gypsum, which, in most cases, are normally consolidated (NC) soil.

2.5.3 Soil collapse potential

Collapse potential is the additional settlement of a foundation, which takes place due to the wetting of soil without any increase in the applied loads, as described by Jinnings and Knight (1975).

They used two different procedures to calculate the collapse potential of soil suing odometer test. The first procedure was the single odometer test, a test in which the soil will be consolidated by using the traditional consolidation test, but the sample is not saturated until the end of the application of 200 kPa of consolidation pressure. After that, the water is added to saturate the sample under the same 200 kPa pressure, and the consolidation test proceeds until the end, as shown in Figure 2.4.

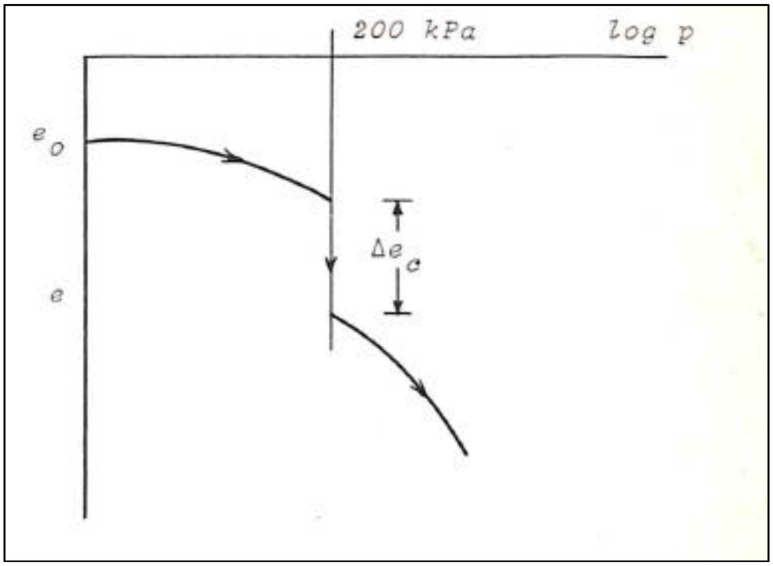


Figure 2.4 Single odometer test (Jinnings and Knight 1975).

The other procedure is the double odometer test. In this test, two odometer tests are done on two samples with the same properties, but one is tested under natural condition (without saturation), the second sample is tested under saturation conditions, as shown in Figure 2.5.

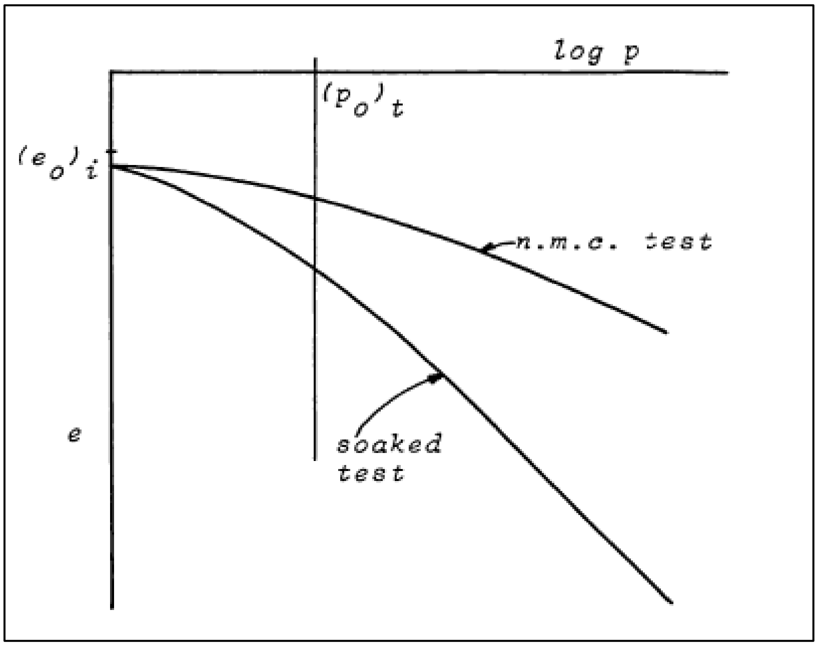


Figure 2.5 Double odometer test (Jinnings and Knight 1975).

The deference between the two procedures is the collapse potential. In the single odometer test, the collapse potential is calculated only under 200 kPa of pressure. However, with the second procedure, it can be calculated under any consolidation pressure. The collapse potential equation: (Jinnings and Knight 1975)

$$CP = \frac{\Delta e}{1+e_o} \times 100 \qquad \text{(Eq. 2.4)}$$

They classified soils according to collapse potential as shown in Table 2.4. As shown in several studies, the collapse potential in soil with gypsum, is highly related to the dissolution of gypsum particles. Mitigating or preventing gypsum dissolution has a big impact in the reduction of the collapse potential.

Table 2.4 Soil classification according to collapse potential.

CP	**Severity**
0-1%	No Problem
1-5%	Moderate Trouble
5-10%	Trouble
10-20%	Severe Trouble
>20%	Very Sever Trouble

2.5.4 Structural problems related to gypsum presence in soil

The main problems in structures caused by the presence of gypsum in soil can be classified as follows:

a- **Subsidence:** The presence of gypsum in soil can cause severe subsidence for structures that are built above this type of soil. This problem is related to the solubility of gypsum in water with time, which is approximately 2.6 g/L.
If the subsidence in soil is relatively high, sinkholes may occur (Elrashidi et al. 2007). Since the solubility of gypsum is highly related to gypsum content, it has been found that gypsum content as low as 1.5% can cause subsidence problems (Nelson 1982). The presence of gypsum as deep as 5 m in the soil keeps this problem in soil due to the movement of water inside the soil from the irrigation system, leakage in water pipes, rainfall, or any source of water infiltration within the soil (Eswaran and Zi-Tong 1991).

b- **Piping:** Piping in soil is a process of dissolving the salts in soil due to moving water, which results in the creation of cavities over time (Maatooq et al., 2014).

By observing deformed buildings in Erevan area in Armenia, Arutyunyan and Manukyan (1982) found that ground water movement caused dissolution and removal of gypsum from the soil over time.

This phenomenon may result in collapse problems in many buildings. Moreover, from site investigations, they detected that the piping problem began at several sites with gypsum content as low as 5%.

Piping related to gypsum rocks dissolution is the main reason behind the occurrence of many sinkholes in the downstream of the Mosul dam in Iraq, as described by Kelley et al. (2007), and Adamo and Al-Ansari (2016).

c- **Corrosion:** Two types of corrosion occur in structures built on soil with gypsum. The sulfate ions affect the main components of the structure (i.e., the reinforcement and the concrete).

Reinforcement corrosion: In a 2009 report by the National Highway Institute (NHI) , they described the process of metal corrosion as the result of electrical current moving from the anodic area to the cathodic area in the soil (the electrolyte).

This process will result in the corrosion of the anodic area due to the transportation of the ions from the metal to the electrolyte. They stated that this problem will happen in soils with high salts concentrations, particularly sulfates, chlorides, and bicarbonates, which make the soil highly acidic or alkaline.

Concrete corrosion: The corrosion of the concrete will result in concrete decay over time through the following reactions that are related to the sulfate ions in gypsum:

- The reaction between the sulfate ion from gypsum and the hydrated calcium aluminate in the concrete to create ettringite [$Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$)] (Shanahan and Zayed 2007, cited by Herrero et al., 2009).
- The reaction between the sulfate ion and the calcium hydroxide during the process of concrete hydration to form gypsum (Tian and Cohen 2000, cited by Herrero et al., 2009).
- The creation of thaumasite [$Ca_3Si(CO_3)(SO_4)(OH)_6\cdot 12(H_2O)$] if the carbonate ion is available due to the reaction between this ion and the sulfate ion (Crammond 2002, cited by Herrero et al., 2009).
- The crystallization process of the sulfate ion in the porous media to create the mirabilite or the sodium sulfate heptahydrate (Hamilton et al. 2008, cited by Herrero et al. 2009).

2.6 Traditional treatment when gypsum is encountered in soil

The traditional approach for dealing with a construction site with gypsum in the soil is to replace it with another type of soil to a specific depth under the shallow foundations. In most cases, the borrowed materials are cheap, have good engineering properties, and are available close to the construction site. This process is governed by the following factors:

- Soil type
- Gypsum content
- Gypsum particle size
- Soil grain size distribution
- Gypsum type (primary or secondary gypsum)

- Depth of the gypsum layer from the ground surface
- Water table
- Type of the structural loads
- The judgment of the geotechnical engineering consultant

All these data are collected from the site investigation, soil test, and the structural design. In most cases, when the soil properties are good, the gypsum content controls the decision to replace the soil.

If the gypsum content is lower than 5%, then no replacement is needed. However, in many cases, this choice is not considered to be the best option, because several observations have shown that cracks and settlement occurs in buildings and houses that have been constructed on soils with gypsum content less than 5%.

Therefore, many consulting engineers choose to replace the soil, even if the gypsum content is low. The steps to replace the soil are shown below:

1. Excavate and remove natural soil from 2 to 3 m larger than the area, Figure 2.6.

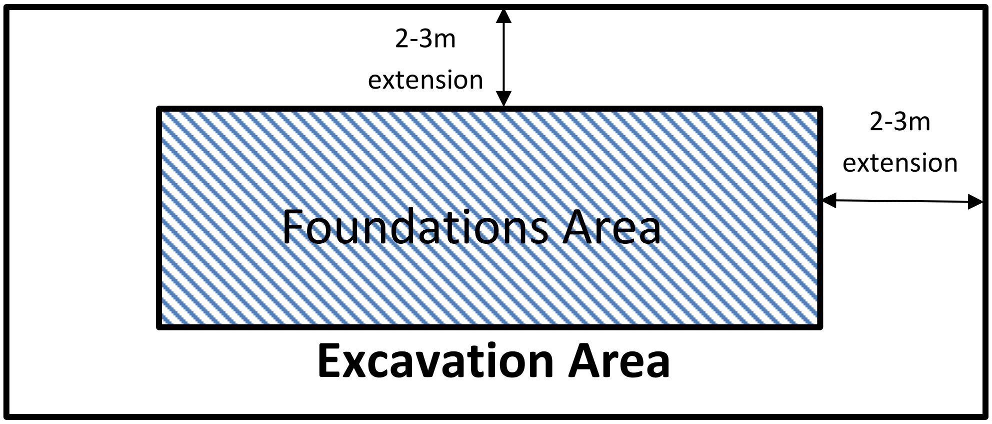


Figure 2.6 Excavation for traditional treatment for soil with gypsum.

2. The depth of soil removal is controlled by the depth of the bearing capacity surface, which may reach up to 10 m.

3. In many cases, the natural soil will be compacted beneath the excavation level.
4. Sub-base material (granular soil) will be added in layers and compacted to a field dry density about 95-98 % of the maximum dry density in the laboratory by the modified proctor method.
5. These layers will be added until the required ground level is reached.
6. The bearing capacity for these bed layers will no more than 200 kN/m^2.
7. Sulfate resistant concrete will be used for all foundations.
8. All water and sewer pipes will be sealed to prevent any future leaks to infiltrate the soil.
9. Septic tanks will be kept as far away from the building as possible.
10. An apron will be built with a width no less than 2 m around the building to prevent surface water from infiltrating the foundation.

2.7 Literature review of soil with gypsum treatment

Different improvement techniques were used to modify the compressibility, collapse potential, and gypsum dissolution of different types of soils with gypsum. Fattah et al. (2012) used dynamic compaction to compact previously remolded samples of poorly graded sand with gypsum (SP – SM).

They compacted the soil in 50 cm x 50 cm x 35 cm box with three different tamper weights (2, 3, and 5 kg). The number of blows ranged between 20 and 40 with drop heights of 35, 50, and 65 cm. They used an odometer test to find the compression index (Cc) before and after compaction.

They concluded that twenty drops provided the best reduction in Cc for three different sandy soils with gypsum content of 27, 41.1, and 60.5 %. Moreover, they found that as the gypsum content increased, dynamic compaction had a significant impact to reduce the soil compressibility.

After soaking the dynamically compacted samples, they showed a reduction in the void ratios by comparing them to samples that were not treated with dynamic compaction, which meant a reduction in soil collapse potential.

The second study, which used the deep dynamic compaction (DDC) method in the field, was conducted by Al-Layla and Al-Saffar (2014). In this work, a tamper that weighed (2.4 metric tons) was used to compact a low plasticity silty clay gypsiferous soil (CL-ML) with gypsum content that ranged from 4.5 to 18.6%.

They also used the odometer test to find the compression indices before and after modification. A range of Cc values for undisturbed samples before and after DDC treatment was provided. Before compaction, the range was 0.20 - 0.24, after compaction, the range was 0.084 - 0.100.

Clinker additive was used by Al-Neami (2010) to modify a poorly graded gypseous sand (SP) with 40% gypsum content. The work was done by using different percentages of clinker (2, 4, and 6%) in remolded samples. The results showed that 4% clinker additive reduced the compression index, Cc, from 0.17 to 0.10.

The same percentage provided a reduction of 73% in the collapse potential. Karim et al. (2012) used commercial bentonite and kaolinite to enhance the physical and mechanical properties of gypseous sandy soil with gypsum content of approximately 50%.

For compacted samples at natural field density, they found that the compression index, Cc, was reduced from 0.149 before treatment to 0.118 after adding approximately 10% of bentonite, and to 0.133 with 10% kaolinite.

Fattah et al. (2013) used acrylate grout to treat four types of silty sand with gypsum content ranging from 18 – 72%. They found that this process had many effects, including increased shear strength, reducing soil compressibility, and reduced collapsibility. The reduction in soil compressibility was approximately 60 - 70 %.

Awn (2011) used pre-wetting to reduce soil collapse potential and the foundation settlement for two types of low plasticity silty soils with gypsum contents of 50 and 70% by using a laboratory model with dimensions of 320 mm x 472 mm and a circular footing that was 50 mm in diameter.

He found that after three cycles of wetting under applied constant stresses of 45 kN/m^2, and 100 kN/m^2, a reduction in the S/B ratio (settlement / footing width) of approximately 63% was achieved for a soil sample with 50% gypsum content, and a reduction of 86% for a soil sample with 70% gypsum content sample. Also, for the soil with 70% gypsum content, the field study with the applied stress of 100 kN/m^2 showed a reduction in the S/B ratio of approximately 90%.

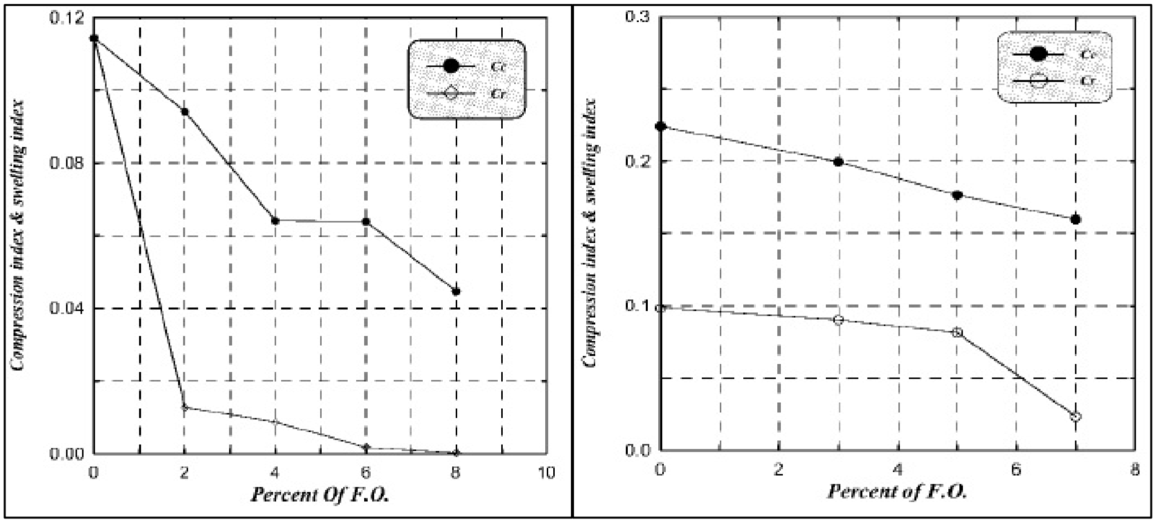
Alsafi et al. (2017) used activated fly ash to immobilize the gypsum in soil to improve its strength and to reduce its collapse potential. The target was to stabilize gypsiferous clayey sand with silt that has a gypsum content of 13%.

The fly ash was activated with different Alkali activators, (NaOH and KOH) which have three different molarities of (8, 10, and 12M). All the treated samples were exposed to sulfate attack using $MgSO_4$ solution.

After comparing the treated samples with samples treated with Portland cement and exposed to same conditions, the results showed that activating the fly ash with 12M of KOH and using 30% of the activated fly ash with the soil will provide the best reduction in the collapse potential and the coefficient of permeability.

Fuel oil was used as a treatment for gypseous soils by Aziz and Ma (2011) by mixing it with two different types of soils: gypseous sandy soil with gypsum content of approximately 52%, and gypsiferous clayey soil with gypsum content of approximately 27%.

They found that mixing 8% of fuel oil with soil will result in many modifications, such as the reduction in soil compressibility. Figure 2.6 shows the reduction in both the soil compression index, Cc, and the swelling index, Cr, for both types of soil.



Gypseous sandy soil Gypsiferous clayey soil

Figure 2.7 Compression and swelling indices modification (Aziz and Ma 2011).

Taha et al. (2008) treated poorly graded gypseous sandy soil (SP) which has gypsum content between 40 – 50 % by using different percentages of cut-back RC-70 (2, 4, 6, and 8%). Two techniques have been used to add the asphalt: by injection and mixing. They found that mixing the asphalt with the soil provided the best result.

Moreover, 6% asphalt provided the most improvement in the dry density, unconfined compression strength, and the shear strength parameters. The collapse potential decreased with an increase in the asphalt percentages.

Chapter 3

Gypsum and Collapse Potential Measurements

3.1 Gypsum content measurements

Three different dehydration methods have been used to calculate the gypsum content for the following natural soils:

- **Soil 1 (S1):** Lark series, which is a white gypseous sandy soil from the Barchan Dune in of New Mexico.
- **Soil 2 (S2):** Hembrillo series, which is a brownish sandy soil with some roots and leaves. It is located at the northeastern end of the White Sands dune field in the New Mexico. Both soils were received from the Natural Resources Conservation Service (NRCS) office in Las Cruces, New Mexico. Figures 3.1 and 3.2 show the grain size distributions.

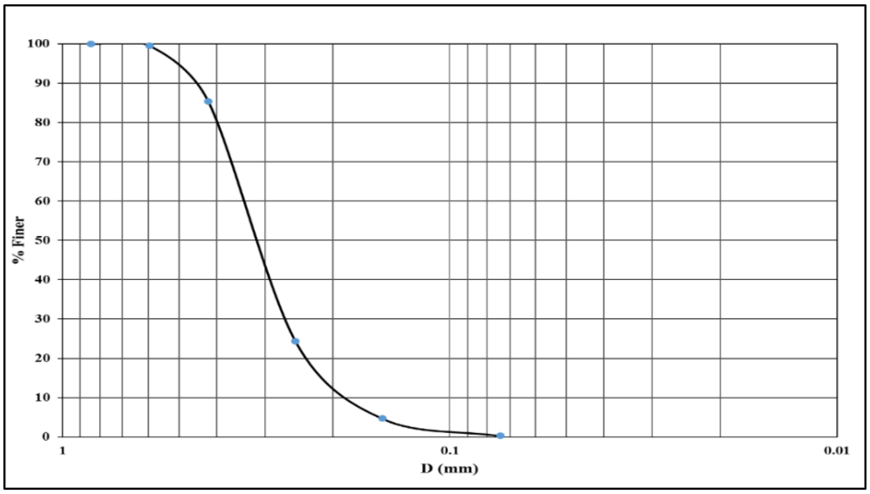


Figure 3.1 Grain size distribution for Soil 1.

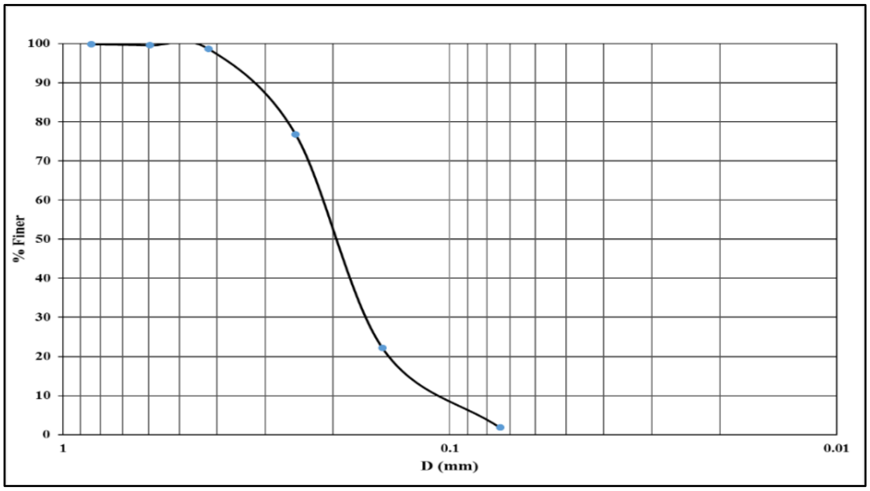


Figure 3.2 Grain size distribution for Soil 2.

The results show that both soils are fine, poorly graded sand, according to the USCS classification.

3.1.1 Al-Mufty and Nashat (2000) method

For this method, the soil sample was dried in the oven at 45ºC (113ºF), to remove the moisture. The weight of the dried sample was Record when the weight reached a constant value. After that, the sample was dried at 110ºC (230ºF). The weight of the sample was recorded again after it reached a constant value (Fattah et al., 2012). The gypsum content was calculated as follows:

$$x\ (\%) = \frac{\mathrm{W45\ ^{o}C - W110\ ^{o}C}}{\mathrm{W45\ ^{o}C}} \times 4.778 \times 100 \qquad \text{(Eq. 3.1)}$$

x = Gypsum content (%)

W45ºC = Weight of the sample at 45ºC

W110ºC = Weight of the sample at 110ºC.

During the test, the weights were recorded when the difference between the last two readings was approximately ±0.02 g.

100 g of air-dried soil samples were used in the test, and the weights were checked every 24hrs. Table 3.1 shows the values of the W110ºC and the gypsum content over time for two samples from Soil 1 (S1).

Table 3.1 W110ºC weights and gypsum content for Soil 1.

Dish. No.	Date	Time (min)	W110ºC	Gypsum %
D1	12/9/2016	1440	84.25	75.09
	12/10/2016	2933	80.47	93.16
	12/11/2016	4342	80.27	94.12
	12/12/2016	5778	80.35	93.73
	12/13/2016	7160	80.44	93.30
	12/14/2016	8573	80.39	93.54
	12/15/2016	10019	80.38	93.59
D3	12/9/2016	1440	83.85	77.04
	12/10/2016	2933	80.61	92.53
	12/11/2016	4342	80.26	94.20
	12/12/2016	5778	80.35	93.77
	12/13/2016	7160	80.4	93.53
	12/14/2016	8573	80.42	93.44
	12/15/2016	10019	80.34	93.82

Figures 3.3 and 3.4 show the relationship between time and the W110ºC weights with the gypsum content for Soil 1. These figures show that at approximately 4000 min., W110ºC weights reached constant values (between 80.25-80.5 g).

At the same time, the gypsum contents also reached constant values (between 93-94 %). The standard deviation for all the values after 4000 min. was approximately 0.43.

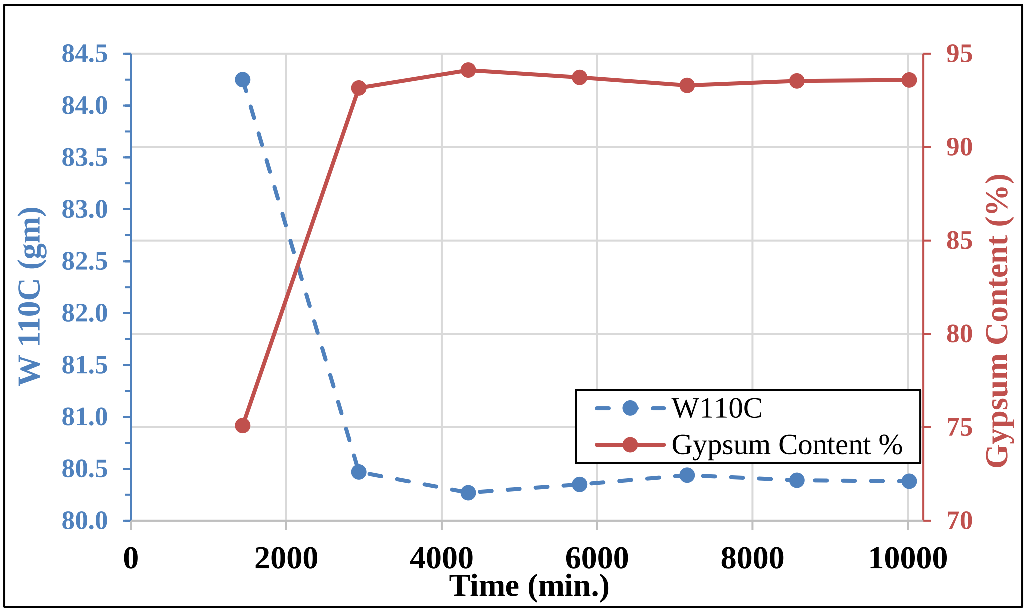


Figure 3.3 Relationship between W110ºC and gypsum content and time for Soil 1/Sample D1.

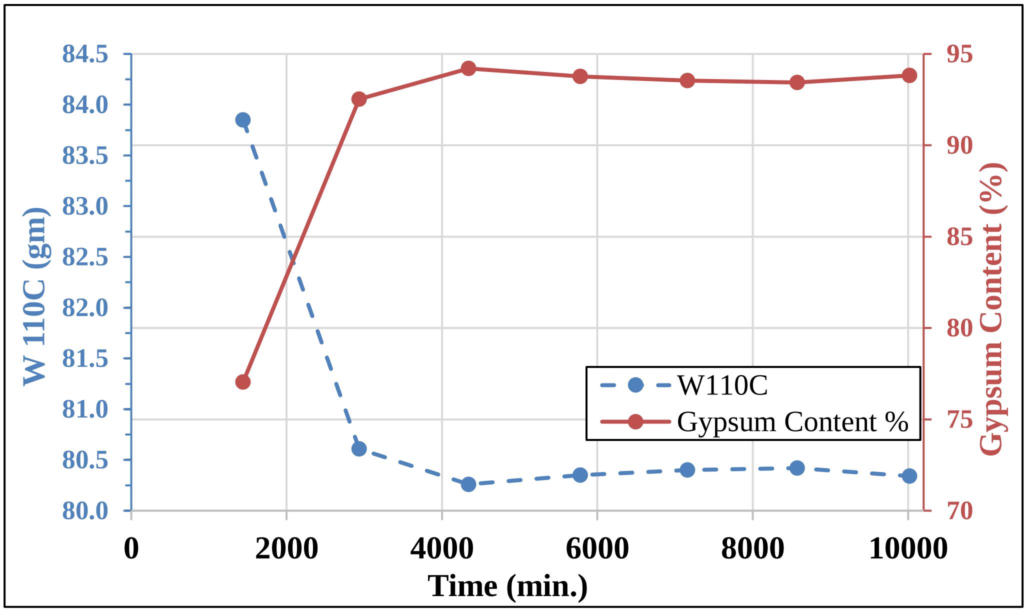


Figure 3.4 Relationship between W110ºC and gypsum content and time for Soil 1/Sample D3.

Table 3.2 also shows the values of W110ºC and the gypsum content over time for two samples from Soil 2 (S2). Figures 3.5 and 3.6 show the relationship between time and W110ºC weights with the gypsum content for Soil 2.

Table 3.2 W110ºC weights and gypsum content for Soil 2.

Dish No.	Date	Time (min)	W110ºC	Gypsum %
D7	12/9/2016	1440	93.57	29.92
	12/10/2016	2933	93.44	30.54
	12/11/2016	4342	93.46	30.44
	12/12/2016	5778	93.5	30.25
	12/13/2016	7160	93.55	30.01
	12/14/2016	8573	93.56	29.96
	12/15/2016	10019	93.49	30.30
D8	12/9/2016	1440	93.25	31.40
	12/10/2016	2933	93.23	31.50
	12/11/2016	4342	93.24	31.45
	12/12/2016	5778	93.26	31.36
	12/13/2016	7160	93.35	30.92
	12/14/2016	8573	93.34	30.97
	12/15/2016	10019	93.28	31.26

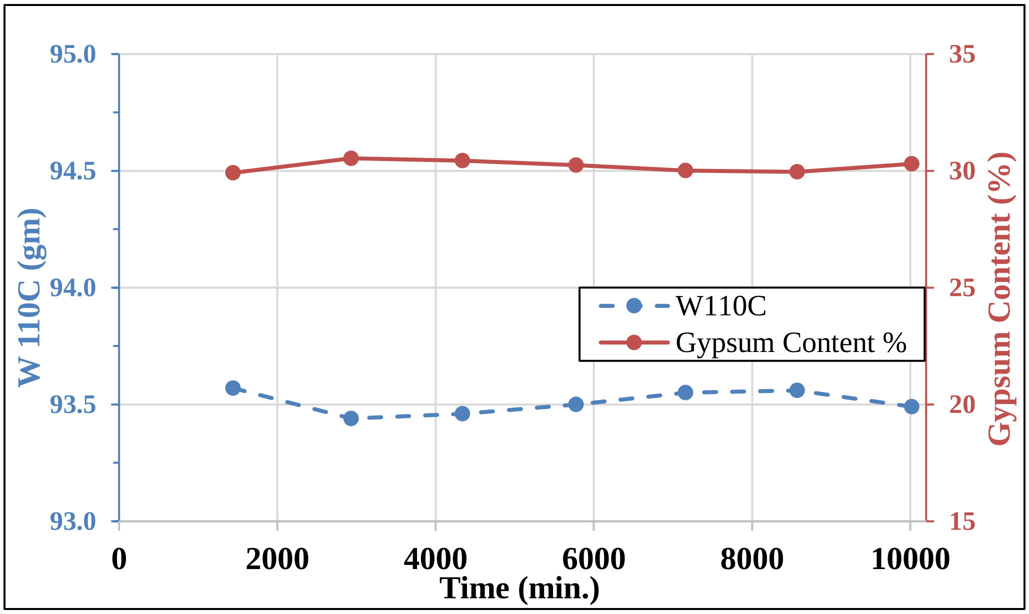


Figure 3.5 Relationship between W110ºC and gypsum content and time for Soil 2/Sample D7.

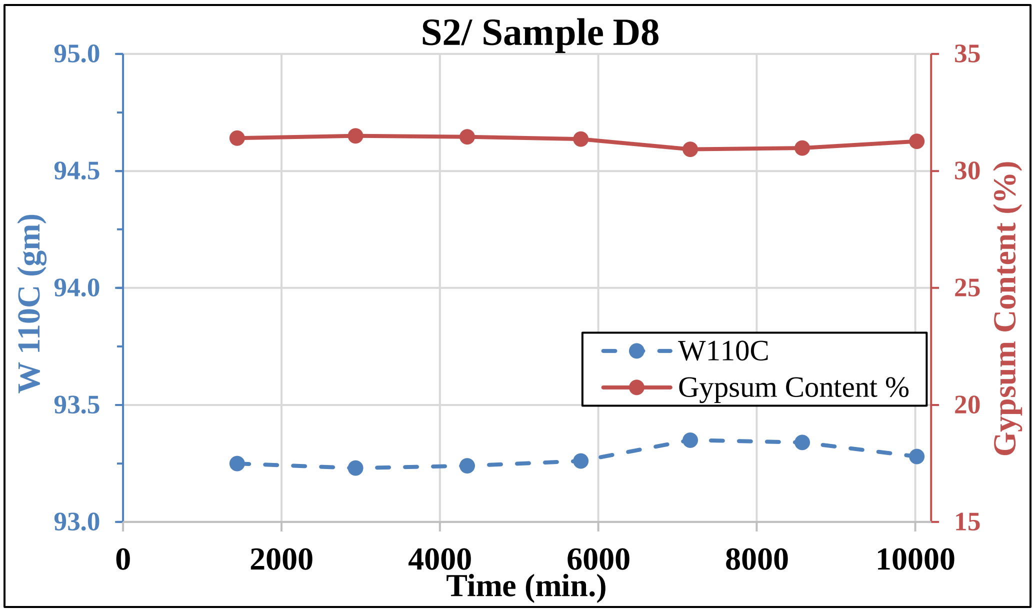


Figure 3.6 Relationship between W110ºC and gypsum content and time for Soil 2/Sample D8.

From the figures, it can be inferred that at approximately 3000 min., the W110ºC weights reached constant values for sample D7 (between 93.38-93.63 gm) and for sample D8 (between 93.24- 93.35 g).

The gypsum content reached constant values for sample D7 (between 30-31 %) and for sample D8 (between 31-32 %). The standard deviation for all the values after 3000 min. is approximately 0.54.

3.1.2 Silica gel method (Nelson et al., 1978)

This method is dependent on the loss of the crystal water in gypsum when the soil is heated at 105°C. At the beginning, the soil sample is dried in a desiccator with silica gel for 48 hrs. and then placed in an oven at 105ºC for 24 hrs.

To calibrate the crystal water in gypsum, pure gypsum was used as a reagent and was also dried in both the desiccator and in the oven at 105ºC, exactly as the soil samples.

Then, the gypsum content was calculated by using the crystal water loss value in an equation to calibrate the gypsum content from the water loss method to the SO_4 method, which is the standard SO_4 method (Nelson et al., 1978).

3.1.2.1 Calibrations

a- The silica gel weight: For this method, the soil sample and the silica gel were placed in a wide-mouth pint mason jar. In this study, a desiccator with an inner diameter of 300 mm was used, which could contain multiple samples during the test. Therefore, due to the difference between the volume of the wide-mouth pint mason jar and the desiccator, the appropriate amount of silica gel that can be placed in the desiccator was determined. Water was used during this calibration to calculate both volumes of the wide-mouth pint mason jar and the desiccator.

- A wide-mouth pint mason jar volume = 473 ml of water.
- The volume of desiccator = 11,960 ml of water.
- The desiccator = 25.3 wide-mouth pint mason jars.
- With this method, for one wide-mouth pint mason jar, 10 g of silica gel was needed. Therefore, for the desiccator, 253 g of silica gel was needed to reach equilibrium.
- After placing the silica gel in the desiccator, vacuum grease was placed on the edge of the desiccator, sealed, and left for 24 hrs. to reach equilibrium.

b- The Moisture Dishes: For this method, an aluminum moisture dish (D = 600mm, depth = 15mm) was needed. This size was not available; therefore, an aluminum dish with D = 50 mm and depth = 21 mm was used.

c- Scale: This method required a scale with sensitivity = 0.001 g, due to unavailability, a scale with sensitivity = 0.01 g was used.

3.1.2.2 Calculations

1- Crystal water content in Gypsum, g/g: According to the procedure, the crystal water in the gypsum was calculated from the following equation

$$Wc = \frac{(wt.3 - wt.4)}{(wt.3 - wt.1)} \qquad \text{(Eq. 3.2)}$$

Wc: Crystal water content in Gypsum, g/g.

wt.1: Dish weight.

wt.3: Dish + desiccator-dry gypsum for 48 hrs.

wt.4: Dish + oven-dry gypsum for 24 hrs.

2- Gypsum %: By the crystal water loss method on an oven-dry wt. basis.

$$Gypsum\ \% = \frac{(wt.3 - wt.4)(100)}{(wt.4 - wt.1)(Wc)} \qquad \text{(Eq. 3.3)}$$

3- Estimated gypsum %: By the standard SO_4 method on an oven-dry wt. basis.

$$Estimated\ Gypsum\ \% = \frac{(wt.3 - wt.4)(96.1)}{(wt.4 - wt.1)(Wc)} - (0.19) \qquad \text{(Eq. 3.4)}$$

4- Estimated gypsum %: By the standard SO_4 method on a soil oven-dry + gypsum crystal-water wt. basis.

$$Estimated\ Gy.\%\ from\ both\ methods = \frac{(Estimated\ Gypsum\ \%)}{1 + (Gypsum\ \%)(Wc/100)} \qquad \text{(Eq. 3.5)}$$

wt.1: Dish weight.

wt.3: Dish + desiccator-dry soil for 48 hrs. and wt.4: Dish + oven-dry soil for 24 hrs.

3.1.2.3 Test procedure and results

Five samples for each soil were used to determine the gypsum content. Tables 3.3 and 3.4 show the value of gypsum contents for Soil 1, and Soil 2.

Table 3.3 Gypsum content for Soil 1.

Dish	**wt1 (gm)**	**wt3 (gm)**	**wt4 (gm)**	**Wc**	**GY %**	**Estimated GY %**	**Estimated GY%**[1]
CH	10.86	18.86	17.33	0.1975	119.7	114.9	92.93
E2	11.13	19.13	17.59	0.1975	120.7	115.8	93.51
E11	11.19	19.19	17.66	0.1975	119.7	114.9	92.93
E1	11.18	19.17	17.65	0.1975	119	114.1	92.39
E6	10.85	18.85	17.31	0.1975	120.7	115.8	93.51

[1] Estimated by both methods

Table 3.4 Gypsum content for Soil 2.

Dish	**wt1 (gm)**	**wt3 (gm)**	**wt4 (gm)**	**Wc**	**GY %**	**Estimated GY %**	**Estimated GY%**[1]
E9	11.12	19.09	18.59	0.193	34.7	33.1	31.02
K12	11.12	19.1	18.62	0.193	33.2	31.7	29.79
E10	11.14	19.11	18.61	0.193	34.7	33.1	31.02
E13	11.19	19.17	18.66	0.193	35.4	33.8	31.64
1	11.19	19.17	18.66	0.193	35.4	33.8	31.64

[1] Estimated by both methods

The results show that Soil 1 has a gypsum content between 92-94%, with a standard deviation of approximately 0.42, whereas, Soil 2 has a gypsum content between 29-32%, with a standard deviation of approximately 0.67.

3.1.3 OMRAN GypSim method (Omran, 2016)

3.1.3.1 Using OMRAN method to calculate gypsum content for natural soils

Gypsum content was calculated using this method by heating the samples at 70ºC (158ºF) for 15 min. and then at 150ºC (302ºF) for 15 min. The gypsum content was determined by using the following equation:

$$Gypsum\ \% = \frac{(W70-W150)}{(W70-Wd)} * 100 * (\frac{100}{19.66}) \qquad \text{(Eq. 3.6)}$$

W70 = weight of the sample dried at 70ºC + Pyrex dish.

W150 = weight of the sample dried at 150ºC + Pyrex dish.

Wd = weight of the Pyrex dish.

19.66 = the recovery factor of gypsum between 70 and 150ºC.

This method required a scale with a sensitivity of 0.001, but this type of scale was not available. Therefore, a scale with a sensitivity of 0.01 was used.

3.1.3.1.1 Test procedure and results

Four samples of each soil were used, with 20 g of air-dried soil. During the first trial, samples were heated at 150ºC for 15 min. (as described in the procedure), but the results were very small compared to previous methods. Therefore, three more trials were used with increased time to find a suitable time to dry all the crystal water in the soil gypsum. During the test, samples were left in the desiccator for approximately 5 min. to cool after drying at 70ºC, and for approximately 10 min. after drying at 150ºC oven before taking the weights. Tables 3.5, 3.6, 3.7, and 3.8 show the four sets of tests for both soils (S1 and S2).

Table 3.5 Set No. 1 (12/27/2016).

Soil	**Dish No.**	**Wd (gm)**	**W70C (gm)**	**W150 (gm) / 15 min.**	**GY %**
S1	C	44.36	64.38	63.96	10.67
	A	42.9	62.9	62.58	8.14
	1	57.68	77.67	77.27	10.18
	6--3	42.78	62.78	62.46	8.14
S2	2	43.54	63.5	63.2	7.65
	3	42.47	62.43	61.96	11.98
	5	51.18	71.12	70.79	8.42
	9--1	50.59	70.56	70.19	9.42

Table 3.6 Set No. 2 (01/20/2017).

Soil	Dish No	Wd (gm)	W70C (gm)	W150 (gm)				GY %
				35 min	55 min	75 min	95 min	
S1	6--3	42.77	62.78	61.41				34.83
	1	57.7	77.66		75.25			61.42
	2	43.53	63.57			61.1		62.69
	3	42.47	62.47				59.5	75.53
S2	A	42.9	62.88	61.98				22.91
	C	44.35	64.31		63.18			28.8
	S	51.18	71.13			69.86		32.38
	9--1	50.59	70.56				69.2	34.64

Table 3.7 Set No. 3 (01/23/2017).

Soil	Dish No	Wd (gm)	W70C (gm)	W150 (gm)				GY %
				115 min	135 min	155 min	175 min	
S1	No.3	42.47	62.48	59.59				73.46
	S	51.18	71.17		68.07			78.88
	No.2	43.55	63.56			60.08		88.46
	No.1	57.69	77.68				74.18	89.06
S2	6--3	42.78	62.76	61.44				33.6
	9--1	50.6	70.53		69.19			34.2
	C	44.36	64.31			62.96		34.42
	A	42.9	62.87				61.52	34.39

Table 3.8 Set No. 4 (01/25/2017).

Soil	Dish No	Wd (gm)	W70C (gm)	W150 (gm)				GY %
				195 min	215 min	235 min	255 min	
S1	6--3	42.79	62.75	59.01				95.31
	9--1	50.6	70.57		66.73			97.81
	C	44.36	64.35			60.42		100
	A	42.9	62.89				58.96	100
S2	No.3	42.48	62.42	61.1				33.67
	S	51.18	71.12		69.77			34.44
	No.2	43.55	63.52			62.16		34.64
	No.1	57.69	77.62				76.27	34.45

Figures 3.7 and 3.8 show the combination between all four sets of tests for both soils S1 and S2 separately.

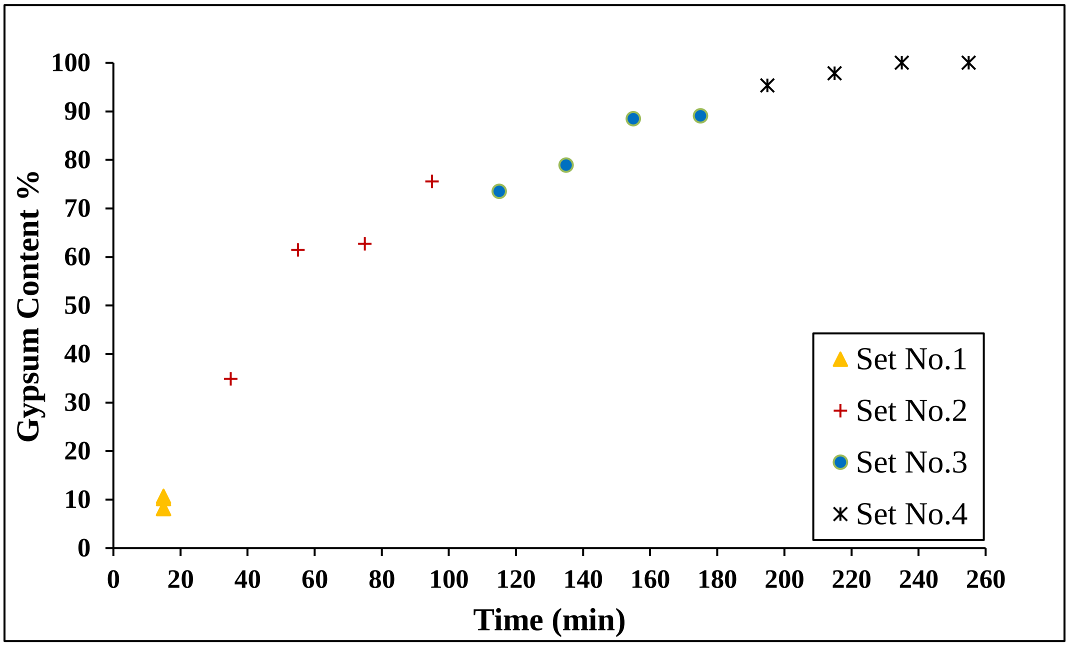


Figure 3.7 The combination of the four sets for Soil 1.

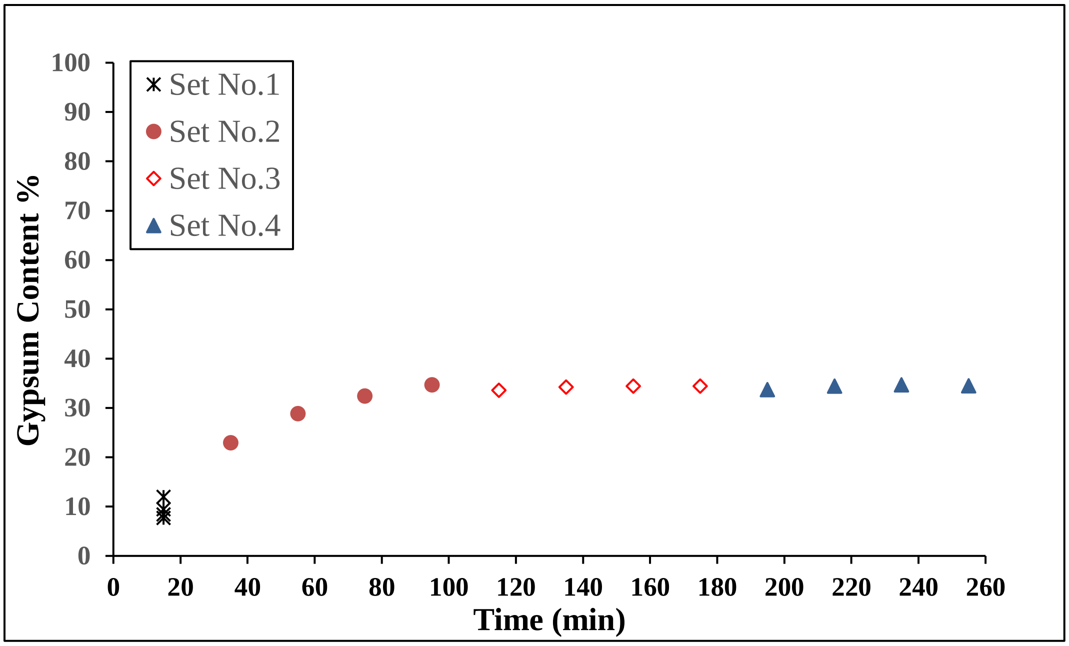


Figure 3.8 The Combination of the Four Sets for Soil 2.

For Soil 1, as shown in Figure 3.7, the gypsum content reach constant values after 215 min. After that time, the gypsum content was 95-100 %, with a standard deviation of approximately 1.03. Soil 2, as shown in Figure 3.8, reached constant values after approximately 115 min., and the gypsum content was 33-35 % with a standard deviation of approximately 0.37.

3.1.3.2 OMRAN method evaluation

Due to the findings obtained using this method, a study was performed to evaluate this procedure. In this work, and in addition to the natural soil samples that were used previously, three other materials were used to calculate the gypsum content at different times with 15 min. intervals. Table 3.9 shows the different materials that were used in this work.

The first material used in this evaluation was a mixture 50% of Soil 1 and 50% of Soil 2. This mixture was used because Soil 1 has an approximate gypsum content of 93%, and Soil 2 has approximate gypsum content of 31%. A mixture of these two soils would produce a soil with approximately 65% gypsum content.

To evaluate a relatively larger gypsum particle, the second material consisted of crushed gypsum rock at different percentages. All-purpose silica sand was used as a filler to achieve desired gypsum content. This soil was a clean, poorly graded sand (SP) manufactured by Quikrete International Inc. (Atlanta, Georgia, USA).

The third material was a synthetic gypsum, which was used to cover smaller gypsum particles. Samples were created by mixing this gypsum with silica sand as a filler.

Table 3.9 Characteristics of gypsum materials.

Gypsum Material	**Gypsum %** [2]	**Particle Size (mm)**	**Gypsum Particle Size (mm)**
Poorly graded fine gypseous sandy soil[1], (S1)[3]	93	0.07 - 0.42	Within the range of soil due to the high gypsum content almost all soil particles are gypsum.
Poorly graded fine gypsiferous sandy soil[1], (S2)[4]	31	0.07 - 0.84	0.42 mm, most of the gypsum passing a sieve number 40
Synthetic gypsum[5]	≥99[7]	Equivalent to poorly graded silt	95 % < 0.1 mm
Gypsum rock[6]	97	0.42- 4.00	0.42- 4.00

[1] According to USCS, and as A-3 soil according to ASSHTO.
[2] According to silica gel method, Nelson et al. 1978.
[3] Brought from Barchan Dune Gypsum, Lark Series, Otero County, New Mexico. Provided by the NRCS office in Las Cruces, New Mexico, USA.
[4] Brought from Hembrillo Series, Sierra County, New Mexico. Provided by the NRCS office in Las Cruces, New Mexico, USA.
[5] Manufactured by Sigma Aldrich Company in St. Louis, Missouri, USA.
[6] Provided by USA Gypsum company located in Denver, Pennsylvania, USA.
[7] According to the manufacturer.

By using these materials to calculate the gypsum content at time intervals of 15 min., a relationship between the relative gypsum content and the real time for different materials was established, which allow the unknown gypsum content for any ground material to be determined, as shown in Figure 3.9. The heating time at 150ºC should be 230 min. or greater.

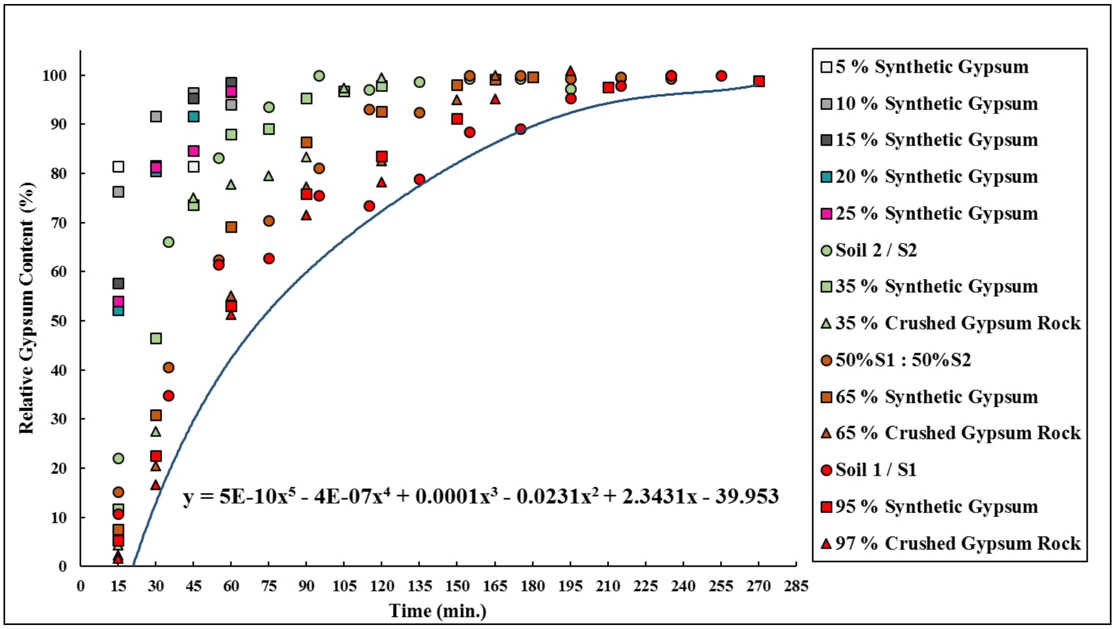


Figure 3.9 Relative gypsum content vs. real time for different ground materials.

3.1.4 Conclusion

From the standard deviations for both soils and from the three methods that were used, the lowest standard deviation for Soil 1was found using method No. 2 (the silica gel method). For Soil 2, the lowest standard deviation was found using method No. 3 (the OMRAN GypSim method).

Based on these results, the best method for high gypsum content is the silica gel method. For low to medium gypsum content, the best method is the OMRAN GypSim method. However, it was found that the OMRAN GypSim method needs more monitoring because it is not clear when the weights at 150ºC stabilize, depends on soil gypsum content. The silica gel method was a straightforward method with a fixed time for drying in both the desiccator and the oven. In conclusion, the silica gel method is the best dehydration method to find gypsum content, and it was adopted for all the future measurements.

3.2 The estimation of the dissolved gypsum in a solution

3.2.1 Electrical conductivity measurement

Based on the concept of the conductance (current transmission ability), an electrical conductivity meter was chosen to estimate the dissolved gypsum in a solution. The meter measures the conductivity based on the presence of ions in the solution.

3.2.2 EC reading calibration

To connect the EC reading to the gypsum concentration in the solution, three different calibrations were performed by using:

- Pure gypsum.
- Natural soil 1 (High gypsum soil)
- Natural soil 2 (Medium gypsum soil)

This calibration was done to find the best formula to convert the EC reading directly into a gypsum concentration to determine the amount of dissolved gypsum. Different solutions were created with different gypsum concentrations, and then the maximum EC reading was taken to establish the relationship between the EC reading and the gypsum concentration.

3.2.2.1 Pure gypsum solutions

Different pure gypsum solutions were created by adding specific amount of pure gypsum to 500 ml of tap water. Then, all the solutions were stirred on a stirring plate, and every 15 min., EC readings were recorded. Once these readings reached a maximum value (a value after which there was no change in the EC reading), the test stopped. At the end of the test, a relationship between the solution concentration and the EC reading was plotted, and an equation was found by using a linear regression method.

Table 3.10 shows the different gypsum concentrations used in the calculation, and Figure 3.10 shows the relationship between the gypsum concentrations and the EC readings.

Table 3.10 Pure gypsum concentrations with EC measurements.

Gypsum Weight (gm)	Water Volume (L)	Concertation (g/L)	EC after first 15 min of stirring (µS/cm)	EC max. (µS/cm)
0	0.5	0.0	137.8	137.8
0.2	0.5	0.4	490.2	494.2
0.4	0.5	0.8	870.2	874.2
0.6	0.5	1.2	1214.2	1217.2
0.8	0.5	1.6	1535.2	1561.2
1.0	0.5	2.0	1758.2	1839.2
1.3	0.5	2.6	1969.2	2150.2
1.5	0.5	3.0	2051.2	2177.2

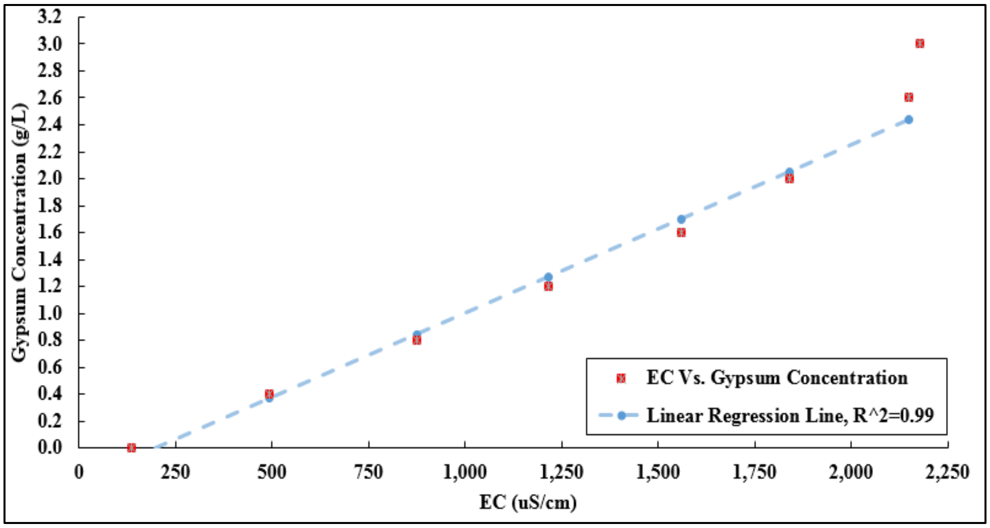


Figure 3.10 Gypsum concentration in pure gypsum vs. EC reading.

Linear regression line equation:

$$\hat{y} = -0.246571068 + (0.001248005 \times x) \qquad \text{(Eq. 3.7)}$$

3.2.2.2 Soil 1 solutions

The previous procedure was conducted with Soil 1 samples. The difference between both procedures, which in this case, the amount of soil that was added would not result in the same amount of gypsum in the solution because even though this soil has about 93% of gypsum content, different soil components make up the remaining 7% of the sample.

Therefore, to find the amount of gypsum that has been added to the water, each amount was multiplied by 93%, as shown in Table 3.11. Figure 3.11 shows the calibration relationship.

Table 3.11 Soil 1 concentrations with EC measurements.

Soil Concentration (g/L)	**Soil Weight (g)/ 500 ml**	**Gypsum Content = 93.05 %**	**EC max. (µS/cm)**
		Gypsum Concertation (g/L)	
0	0.0	0.00	140
1	0.5	0.93	940
1.5	0.75	1.40	1350
2	1.0	1.86	1580
2.6	1.3	2.42	1960
3	1.5	2.79	2090

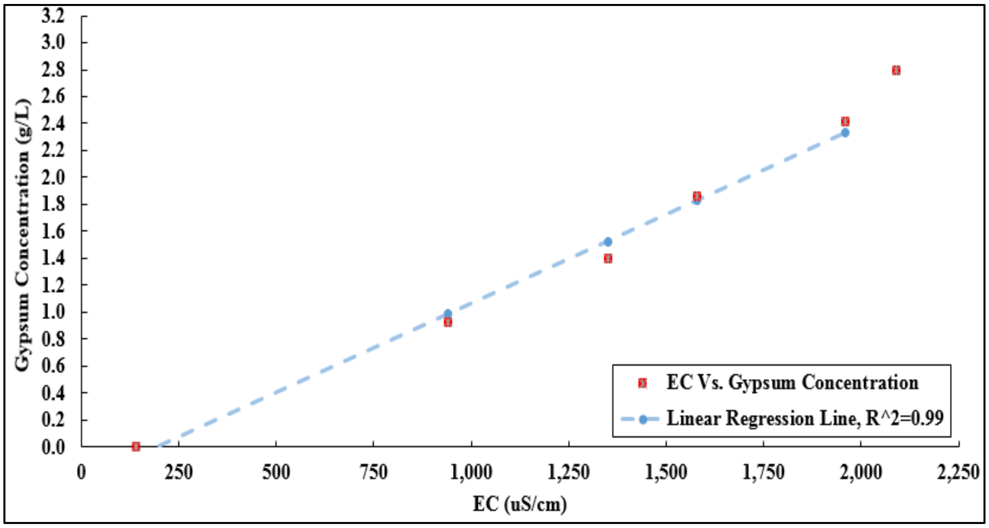


Figure 3.11 Gypsum concentration in Soil 1 vs. EC reading.

Linear regression line equation:

$$y^{\wedge} = -0.253556827 + (0.001318984 \times x) \qquad \text{(Eq. 3.8)}$$

3.2.2.3 Soil 2 solutions

The procedure that was used for Soil 1 was also performed on Soil 2. However, in this case, the soil concentration was multiplied by 31%, which is the gypsum content for Soil 2, as shown in Table 3.12 and Figure 3.12.

Table 3.12 Soil 2 concentrations with EC measurements.

Soil Concentration (g/L)	Soil Weight (g)/ 500 ml	Gypsum Content = 31.02 %	EC max. (μS/cm)
		Gypsum Concertation (g/L)	
0	0.0	0.00	130
1	0.5	0.31	410
1.5	0.75	0.47	570
2	1	0.62	730
2.6	1.3	0.81	900
3	1.5	0.93	1010
4	2	1.24	1250
5	2.5	1.55	1480
6	3	1.86	1690
7	3.5	2.17	1850
8	4	2.48	1980
10	5	3.10	2130

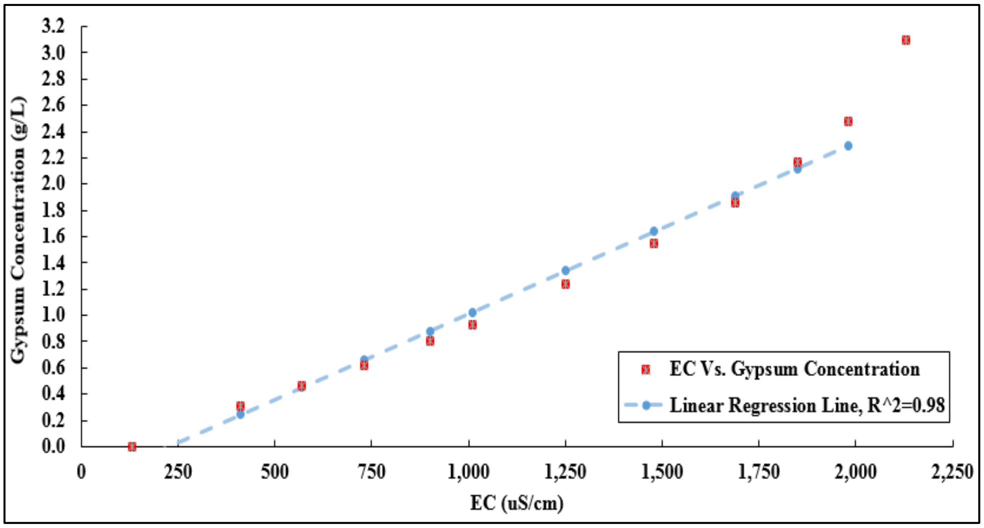


Figure 3.12 Gypsum concentration in Soil 2 vs. EC reading.

Linear regression line equation:

$$y^{\wedge} = -0.290206567 + (0.001302608 \times x) \quad \text{(Eq. 3.9)}$$

3.3 Collapse potential for natural soils

A Jeo Jac odometer device was used, following the procedure for the single odometer test, according to Jinnings and Knight (1975), with 2.5-in. consolidation cell.

3.3.1 Measurements

Two density states, loose and dense, were used during the test for both soils. The loose state was created by pouring the soil in the cell from a distance of 15 cm. The dense state was created by compacting the soil in the cell in three layers with 55 blows per layer using a steel rod.

The test started with a dry condition until the pressure of 200 kPa, and then the testing stopped to add the water. After the addition of the water, the test proceeded from the 200 kPa until the end of the loading. Two or three different amount of pressures were used after 24 hrs. of applying the wet 200 kPa because only the difference in void ratio before and after adding the water was needed. The collapse potential for each case was calculated, as shown in Figures 3.13 and 3.14.

3.3.2 Results and conclusion

1- Moderate to no problem collapse potential was found for both soils.
2- The tests performed in static water conditions with total water volume of 0.45 L (the amount of water filling the consolidation cell) for a period of 24 hrs. The gypsum dissolution measurements showed that the amount of gypsum which has been dissolved was very small and almost negligible.

3- Due to the finding that static water in this type of collapse potential system has no impact on gypsum dissolution, a decision was made not use this system to test the collapse potential for treated samples.

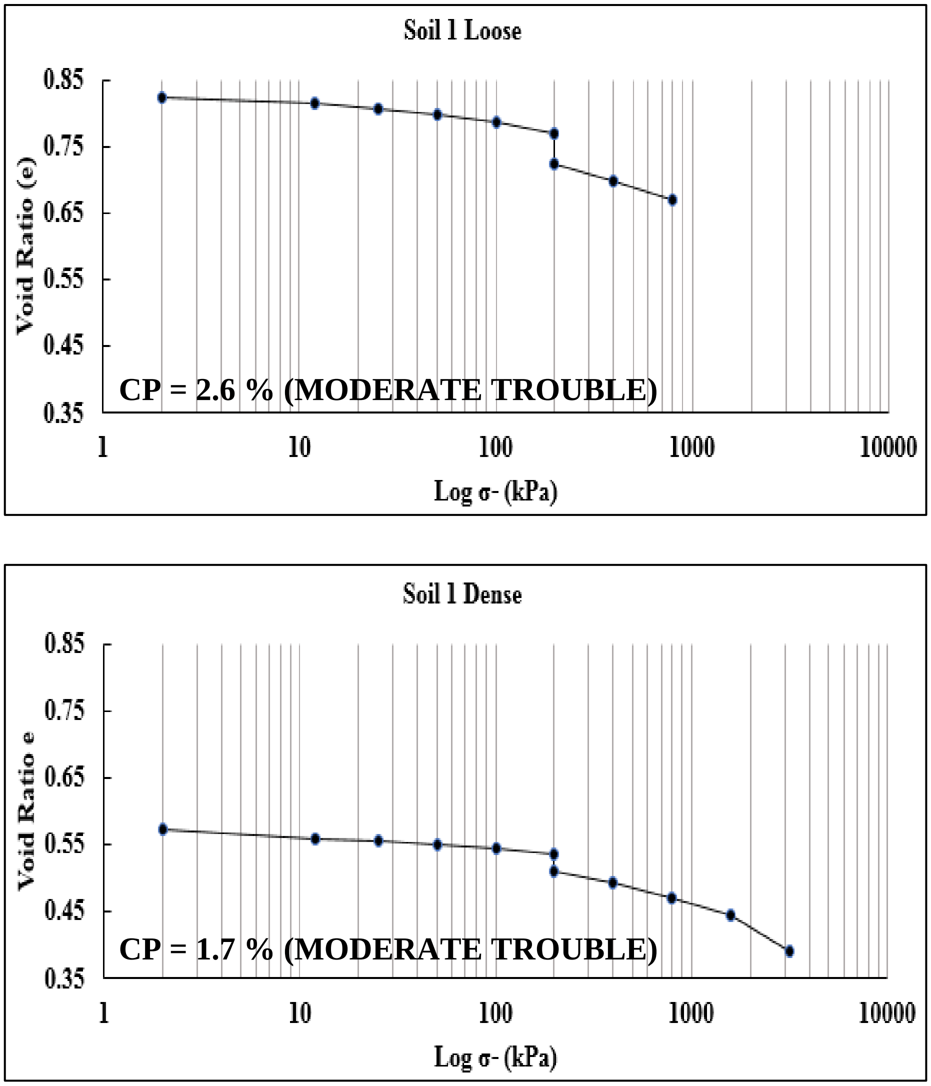


Figure 3.13 Collapse potential for Soil 1.

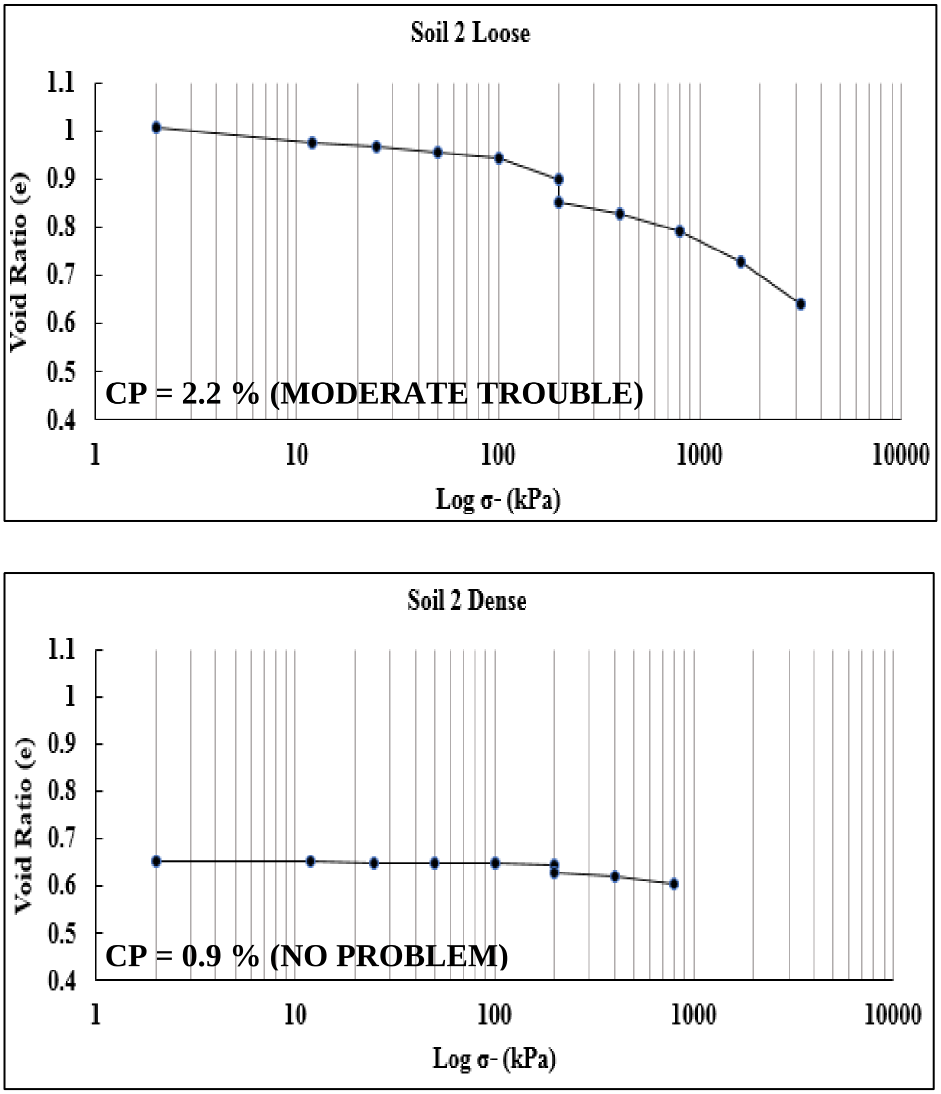


Figure 3.14 Collapse potential for Soil 2.

Chapter 4

The Behavior of Gypseous and Gypsiferous Sandy Soils Treated with Activated Fly ash after Exposed to Wetting-Drying Cycles

4.1 Introduction

Many types of treatments were used to prevent or mitigate the deterioration of soils with gypsum. These methods were based on the type of soil, the gypsum content, particle size, and specific properties to be targeted. Chemical treatment methods represent one of the treatment options, which uses different types of additives, such as geopolymers.

In concrete, the use of a geopolymer (specifically activated fly ash) as an additive to ordinary Portland cement (OPC) to enhance various concrete properties was evaluated in different studies. Saraswathy et al. (2003) used it to improve the corrosion resistance and strength of ordinary concrete. Other studies used it as an alternative to OPC to produce an environmentally friendly concrete and reduce greenhouse emissions (Hardjito et al., 2004; Fernandez-Jimenez et al., 2006; Assi 2018).

Geopolymer paste can be produced using a high-alkaline solution to dissolve the silicon and the aluminum atoms in a source with these materials, such as fly ash (Fernandez-Jimenez et al. ,2006). This process can be enhanced through curing and heating (Hardjito et al., 2004).

It has been found that these materials have excellent mechanical properties, resist acidic attacks, and do not have produce alkali-aggregate reactions, even when in contact with materials with high alkalinity (Davidovits 1999 cited by Hardjito et al., 2004). Using this paste, rather than cement paste, as a binder will hold other unreacted materials (fine and coarse aggregates) together to form geopolymer concrete (Hardjito et al., 2004).

For soil treatment, there are a few studies that discuss the use of geopolymer as an additive to improve some mechanical properties. Abdullah et al. (2017) investigated the improvement of the dry density and strength of kaolin clay stabilized with a combination of granulated blast furnace slag (GBFS), with Class F fly ash activated by Grade D sodium silicate and 14 M concentrated sodium hydroxide (NaOH). They found that using these additives enhanced the maximum dry density, and for curing periods of 7 and 28 days, the strength was equivalent to the use of 9% OPC. For curing periods of more than 28 days, it passed the strength of 9% OPC.

The use of 10, 20, 30, and 40% Class F fly ash activated by 12 M potassium hydroxide (KOH) enhanced the unconfined compressive strength for clayey soil (the highest strength achieved by using the 40%) as was shown by Elkhebu et al. (2018).

In a study by Rios et al. (2016), low calcium fly ash activated by sodium silicate and sodium hydroxide (NaOH) was used to improve the strength, stiffness, and the wetting-drying resistance for silty sand soil to investigate its use in low coast unpaved roads in Colombia. The results indicated an increase in strength and stiffness, and the wetting-drying resistance behavior matched that of the same soil treated with cement.

Geopolymer was also used in limited studies to promote various properties for soils that contained gypsum. A study by Alsafi et al. (2017) used Class F fly ash activated by two different alkali, sodium hydroxide (NaOH) and potassium hydroxide (KOH), at three different molarities (8, 10, and 12 M), to treat clayey sand soil with a gypsum content of 13.2%. To investigate the effects of higher gypsum content, the natural soil was mixed with pure gypsum to make samples with 25% and 45% gypsum content. Curing periods of 7, 28, and 90 days were used to cure the treated samples.

They found that activation with 12 M KOH and 30% activated fly ash provided the greatest improvement in different soil properties, including compressive strength and sulfate attack resistance, along with the reduction in both collapse potential and the coefficient of permeability.

A study by Jha and Sivapullaiah (2017) used 10, 20, and 30% of Class F fly ash as an additive to a mixture of high plasticity clayey soil (CH) and Hydrated lime [$Ca(OH)_2$], along with a range of gypsum (1-6%), to prepare different samples that have been cured for 28, 90, 180, and 365 days.

The findings of this work can be listed as following: the added gypsum and fly ash resulted in the reduction of the plasticity and the shrinkage of the lime-CH mix, an increase in the dry unit weight with fly ash and gypsum increase, and longer curing periods for the samples treated with 30% fly ash provided the highest strength due to the creation of the cementitious compounds (CSH, CAOH, CASH, CAH, and CASHH) as was seen from the micro- analysis (XRD and SEM). While few studies dealt with the use of the activated fly ash with soil that includes gypsum, none of them discussed the use of this additive to treat granular soil with very high gypsum content.

Furthermore, no studies were found that focused on the impact of this additive on gypsum dissolution, which is the most important property for soil with gypsum because this property is linked directly or indirectly to the deterioration of soil properties.

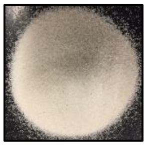
Therefore, this study was designed to investigate the use of activated fly ash to treat poorly-graded sandy soils with 31% and 93% gypsum content and to study the impact of 10, 20, and 30% activated fly ash on volume stability, soil loss, gypsum dissolution, and the unconfined compressive test for specimens cured for 7 and 28 days.

4.2 Materials and methods

4.2.1 Soil samples

Two types of soils were used. Soil 1 (S1) was high gypsum soil, Lark series, which is a white gypseous sandy soil from the Barchan dune in New Mexico. Soil 2 (S2) was medium gypsum soil, Hembrillo series, which is a brownish sandy soil with some roots and leaves. It is located at the northeastern end of the White Sands dune field in New Mexico. Both soils were sent from the NRCS office in Las Cruces, New Mexico. Table 4.1 shows the physical properties of the soils.

Table 4.1 Physical Properties for Soils.

No	**Classification[1]**	**Color**	**Gypsum %[2]**	**Particle Size (mm)**	**Gypsum Particle Size (mm)**
1	Poorly graded fine gypseous sand		93	0.07 - 0.42	Within soil range due to the high gypsum content, (almost all the soil particles are gypsum)
2	Poorly graded fine gypsiferous sand		31	0.07 - 0.84	0.42 mm, most of the gypsum passing a sieve number 40

[1] According to USCS, and as A-3 soil according to ASSHTO classification.

[2] Silica gel method, Nelson et al. 1978.

Moreover, a clean sand (sterling sand), which matches the gradation of these soils, was used to prepare control specimens that do not contain gypsum.

4.2.2 Pure gypsum

Synthetic gypsum was used as the pure gypsum in the test. It was manufactured and purchased from Sigma Aldrich (St. Louis, Missouri, USA). This gypsum had a gypsum content ≥99%, according to the manufacturer, and 95% of the particles are < 0.1mm, which makes its classification equivalents to poorly graded silt.

4.2.3 Fly ash and potassium hydroxide

The fly ash used in this work is classified as Class F, according to ASTM C618-19 and AASHTO M295-19. It was obtained from Cross Generating Station in Pineville, South Carolina, USA. 81% of the sample pass sieve #325, with loss on ignition of 1.8%. 12 M potassium hydroxide (KOH) solution was prepared to activate the fly ash. The KOH was purchased from Sigma Aldrich (St. Louis, Missouri, USA) and has a flaky particle shape.

4.2.4 Specimen preparation and curing method

10, 20, and 30% activated fly ash by dry weight was mixed with dry soil to prepare the specimens. The activation process ensured fly ash:KOH solution ratio (solid: liquid) of 1.2 (Alsafi 2017). Fly ash was mixed with the dry soil by hand for five minutes, then the KOH solution was added and mixed thoroughly with the dry ingredients for approximately ten minutes or until the mixture reached a uniform moisture distribution.

The mixture was divided into three portions and compacted in three layers inside a PVC mold with a diameter and height of 5.0 and 10.0 cm using a small compacting rod. The specimen was then removed from the mold with a manual jack.

The mixture was weighted and measured by taking the average diameter of three measurements and the average height of three measurements. Then, it was sealed with plastic wrap and placed in a moist container.

For curing purposes, a plastic container was used by placing concrete blocks inside and then filling with tap water until ¾ of its volume. Then, a metal net was placed at the top of the blocks to ensure that the specimens would not be in direct contact with the water. The prepared specimens were placed inside the container and then covered with the lid.

Two different sets of specimens were prepared to be cured for two different periods, 7 and 28 days. After curing, the specimens were taken up from the container, the plastic wrap was removed, and they were weighted and measured. After this step, the specimens were ready for the wetting-drying test.

4.2.5 Gypsum content method

A dehydration method called the silica gel method (Nelson et al., 1978) was used to determine the gypsum content of the soil and the remaining specimens. It is based on the concept that the calculation of gypsum is related to the amount of the two chemically bond molecules of water that evaporate when the sample heated at 105ºC for 24 hrs.

4.2.6 Wetting-drying test

The ASTM D559/D559M – 15 standard was used for the test. This standard was designed to determine the mass loss, water content, and the volume change for soil-cement treated samples. Due to the soils' limitation and the presence of gypsum, many deviations have been made to use this standard on soil treated with activated fly ash.

The modifications are listed in Table 4.2. For each percentage of activated fly ash, two specimens were molded, N1 and N2. N1 specimens were used to monitor the changes in volume and water content, while N2 specimens were used to measure mass loss.

Table 4.2 Deviations from ASTM D559/D559M – 15.

Step	**Standard**	**Modification**	**Rational**
Mold	4 X 4.5 in.	2 X 4 in.	Limited soil
Wd in W/C, cys.	Original oven-dry mass	Dry weight after each cycle	Eliminate gypsum dissolution weight
Wetting	5 hrs. continuous	5 hrs., remove samples each hr.	EC measurement
	No water changes	Water changes	No solution saturation
Drying	71º C, 42 hrs.	35º C, 3 days Min.	Constant weight
N2, strokes	4 each end, 20 side	One each end, 9 side	Smaller specimen
N2 Mass loss	A= oven dry mass, 110º C	A= oven dry mass, 35º C	No gypsum dehydration

During the wetting process of each cycle, the electrical conductivity of the solution was measured. The electrical conductivity meter used in this work was a high range EC/TDS meter with a model No. HI99301, which was purchased from HANNA Instruments (Smithfield, Rhode Island, USA). The EC range was between 0.00 to 20.00 mS/cm (0.00-20,000.00 µS/cm) with an EC resolution of 0.01 mS/cm (10 µS/cm).

During the wetting process, the specimens were removed from the water each hour, and the time was stopped when the EC was measured. Afterwards, they were returned to the water until all the five hours of the wetting process ended. The EC measurements were used to estimate the amount of gypsum that dissolved during and after the wetting process. To estimate the amount of dissolved gypsum, different pure gypsum solutions were created by adding specific amounts of pure gypsum to 500 ml of tap water.

Then, the solutions were stirred on a stirring plate, and the EC readings were recorded every 15 min. When these readings reached a maximum value, the test was complete. At the end of the test, the relationship between the solution concentration and the EC reading was plotted, and an equation was found using the linear regression method (Eq. 4.1).

$$\boldsymbol{y}^{\wedge} = -\mathbf{0.25} + (\mathbf{0.0012} \times \boldsymbol{x}) \qquad \text{(Eq. 4.1)}$$

$\boldsymbol{y}^{\wedge}$ = Gypsum concentration (g/L)

$\boldsymbol{x}$ = The electrical conductivity of the solution (µS/cm)

This equation was used to estimate the gypsum that dissolved in each cycle.

4.2.7 Unconfined compressive strength (UCS) test

The procedure in the ASTM D2166/D2166M – 16 standard was used to measure the unconfined compressive strength for the specimens that survived 12 cycles and the control specimens (specimens prepared with the same conditions of the survived specimens but did not go through wetting-drying cycles). A Jeo Jac, an automated device, was used in the test, with a strain rate of 1%/min. (1mm / min.). The Young's modulus (E) was estimated from the stress-strain relationship for each specimen (between 30%-70% of the maximum strength to ensure the measuring value was within the elastic zone).

4.3 Results and discussion

Twenty-four samples with average dimensions of 5. 0 × 10.0 cm were molded to cover 7 and 28 days of curing, as shown in Table 4.3. In this table, high and medium gypsum soils were represented as S1 and S2, F% represented the percentage of activated fly ash that was used to prepare the N1 and N2 specimens. High gypsum soil had more loss compared to medium soil, which mostly occurred during the wetting stage.

Table 4.3 Soil Specimens’ Identification

Specimen ID	Cycles survived	Lost
High gypsum soil		
7 days curing		
S1 F10% N1	None	W of the 1st
S1 F10% N2	None	Removal
S1 F20% N1	4	W of the 5th
S1 F20% N2	None	W of the 1st
S1 F30% N1	6	W of the 7th
S1 F30% N2	1	W of the 2nd
28 days curing		
S1 F10% N1	1	W of the 2nd
S1 F10% N2	2	W of the 3rd
S1 F20% N1	8	W of the 9th
S1 F20% N2	1	W of the 2nd
S1 F30% N1	12	
S1 F30% N2	6	W of the 7th
Medium gypsum soil		
7 days curing		
S2 F10% N1	None	W of the 1st
S2 F10% N2	None	Removal
S2 F20% N1	3	W of the 4th
S2 F20% N2	None	Removal
S2 F30% N1	12	
S2 F30% N2	12	
28 days curing		
S2 F10% N1	None	W of the 1st
S2 F10% N2	1	W of the 2nd
S2 F20% N1	12	
S2 F20% N2	1	W of the 2nd
S2 F30% N1	12	
S2 F30% N2	12	

Two of the specimens fell apart while being removed from the mold. Moreover, the brushing process for N2 specimens exacerbated their deterioration, and only a few of them survived the 12 cycles.

4.3.1 Volume stability

The volume of each specimen was measured after removal from the mold, after curing, and after the wetting and drying processes for each cycle. The percentage of volume change to the initial volume (specimen's volume after removal from the mold) was calculated for each specimen.

Figures 4.1 and 4.2 show the volume changes for two sets (7 and 28 curing periods) of high and medium gypsum soil specimens that were treated with 20% and 30% activated fly ash. None of results for 10% treated specimens have been listed because many of them were lost during early cycles (i.e., none of them passed the wetting part of the third cycle).

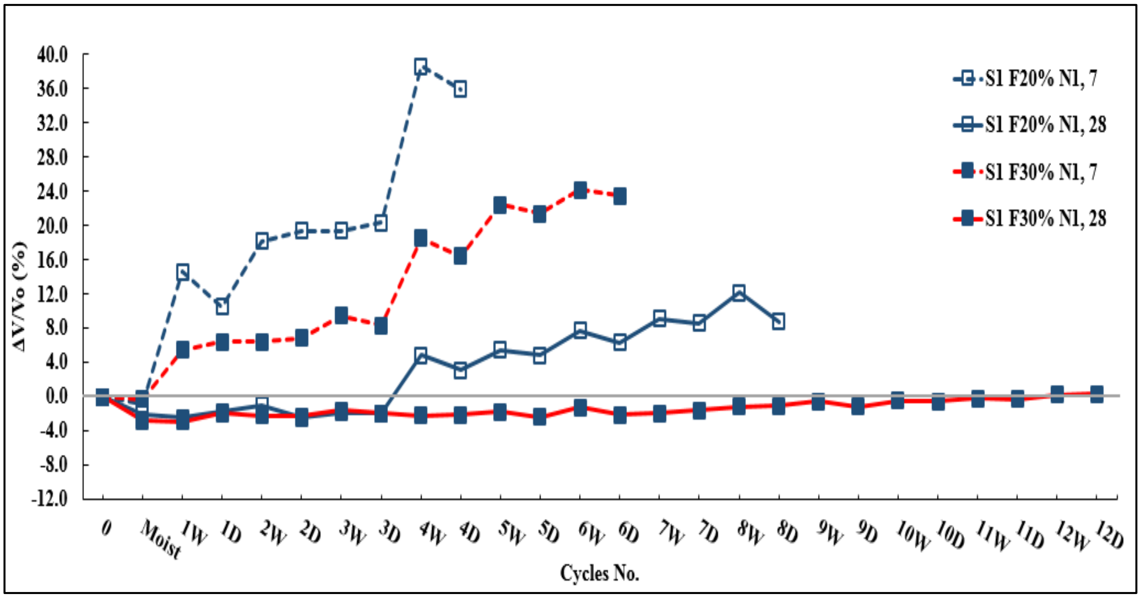


Figure 4.1 Effect of activated fly ash percent and curing time on volume change stability for high gypsum soil.

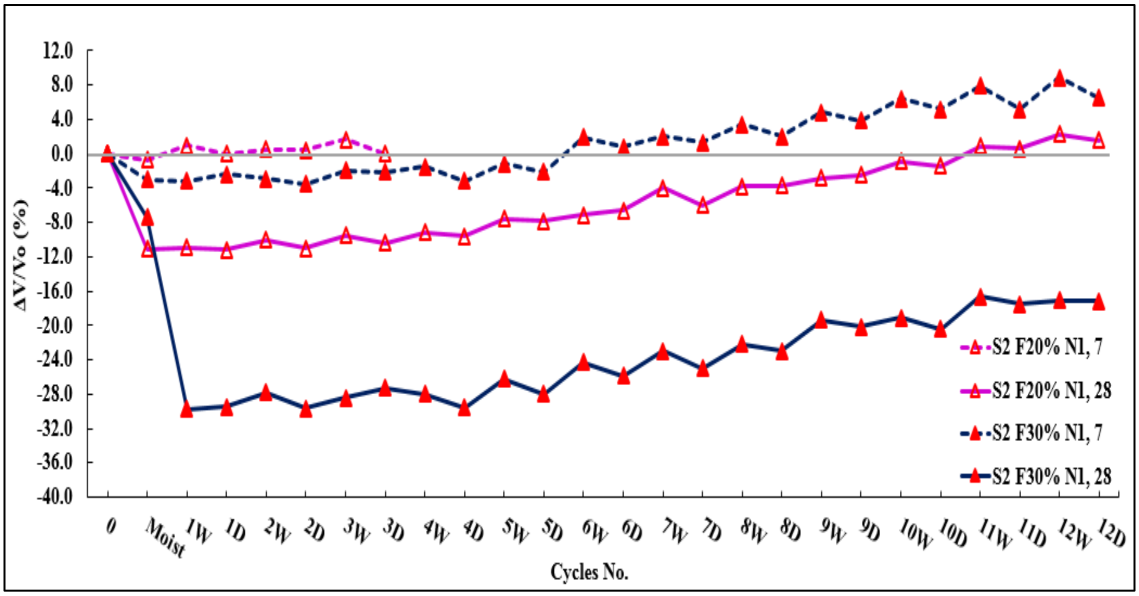


Figure 4.2 Effect of activated fly ash percent and curing time on volume change stability for medium gypsum soil.

The results showed that all the specimens shrank with increasing cycles, However, medium gypsum specimens showed relatively high expansion during the early transition from the moist stage (curing) to the wetting process of cycle one.

The high gypsum soil had very little expansion during curing. This behavior is related to the formation of the ettringite $((CaO)_3(Al_2O_3)(CaSO_4)_3\cdot 32H_2O)$, which filled the voids within the soil's structure. However, when the voids are almost full, there are no more spaces for the ettringite, which will result in an increase in soil volume (expansion) during the curing stage due to the continuous formation of the ettringite (Jha and Sivapullaiah, 2017).

Moreover, the results indicate that more volume stability (less volume change) was achieved with the increase in activated fly ash and the curing period. Previous studies found that the use of fly ash geopolymers enhances different soil properties.

Abdullah et al. (2017) enhanced the dry density and the strength of clayey soils by stabilizing it with different percentages of a mixture of fly ash geopolymer and ground granulated blast-furnace slag (GGBFS). The results showed that the dry density and strength of the soil increased with the increase in the fly ash geopolymer and the curing time.

For soil with gypsum, Alsafi et al. (2017) found that treating silty sand, which has a gypsum content of approximately 13%, with activated fly ash as a geopolymer enhanced the sulfate attack resistance, enhanced the strength, reduced the collapse potential, enhanced soil durability, and reduced the coefficient of permeability. These properties improved with the use of high percentages of activated fly ash and with more curing time.

These improvements in different types of soils when treated with geopolymers (activated fly ash specifically) are related to the formation of calcium-silicate-hydrate(C-S-H) and calcium-aluminate-silicate-hydrate (C-A-S-H) gels and ettringite, as shown by Jha and Sivapullaiah (2017).

They also mentioned that these “cementitious compounds” change the structure of the soil by making it more condense and hardened by creating a reinforced structure with bonding forces that hold the soil particles and fill the voids.

However, it can be seen that with the increase in the wetting-drying cycles, soil shrinkage behavior increased. Moreover, comparing both soil results demonstrates that specimens with high gypsum content suffered from high soil shrinkage, compared to those with medium gypsum content.

This behavior is controlled by the loss of a portion of gypsum particles (which did not react with the geopolymer) by dissolution upon wetting, and because high gypsum soil has more gypsum content than the medium gypsum soil, it showed more shrinkage with cycles. These findings were supported by Aldaood et al. (2014).

In their study, they mixed low plasticity clay (CL) with three different percentages of synthetic gypsum (5%, 15%, and 25%). The mixtures were then treated with 3% lime. They found that after subjecting the specimens to six cycles of wetting-drying, the volume change (shrinkage) increased with cycles, and they even started to deteriorate after the third cycle. These changes were related to the dissolution of the remaining gypsum particles that did not react with the lime and the formation of cracks and ettringite in the soil specimens.

4.3.2 Mass loss

For mass loss calculations, none of the N2 high gypsum soil specimens reached the end of the 12 cycles for the 7 and 28 days curing periods. For medium soil, only N2 specimens treated with 30% activated fly ash finished all 12 cycles. However, both N2 specimens cured for 7 and 28 days showed very high percent of mass loss.

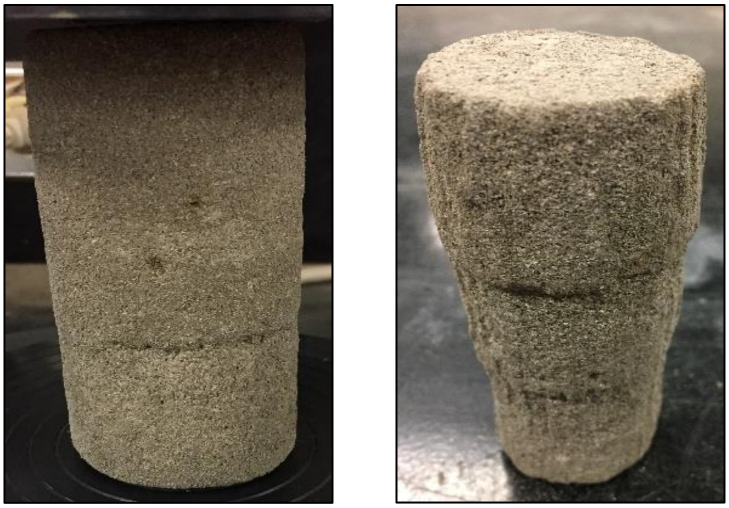
The mass loss for the 7-day cured N2 specimen was approximately 45.82%. For the 28-day cured specimen, the mass loss was approximately 46.16%, almost half of the specimen was lost. There is no mass loss criteria listed in the ASTM D559/D559M – 15 standard. For the few studies on cement-treated soils, the highest mass loss was approximately 19% for a granular soil with the classification of A-1-b, as listed in the standard. However, this value still cannot be used as a reference to judge the mass loss.

From the observations of all specimens that fell apart during the wetting process of different cycles, it was very clear that the core of the specimen was harder than the outside parts, as shown in Figure 4.3. This behavior supports the idea that the activation process of activated fly ash strengthens the soil structure by forming of C-S-H gel, C-A-S-H gel, and ettringite, which has a reverse impact, particularly for long curing periods. This cementitious component was responsible for the deterioration of the specimens, due to post expansion that is related to the ettringite formation (Jha and Sivapullaiah, 2017).

S1 F20% N1 S1 F30% N1

Figure 4.3 Mass loss in high gypsum N1 specimens cured for 7 days.

The high mass loss was also related to the dissolution of gypsum during the wetting part of each cycle. This observation was also found by Aldaood et al. (2014) in their study on lime treated clayey soil with gypsum during the wetting-drying cycles. Figure 4.4 provides a comparison between N1 and N2 medium gypsum soil specimens that were treated with 30% activated fly ash and cured for 28 days after 12 cycles. Although the N2 specimen was brushed 12 times and lost roughly half of its mass, it survived the cycles due to its harder core structure.



N1 after 12 Cycles
N2 after 12 Cycles

Figure 4.4 Medium gypsum soil’s specimens treated with 30% activated fly ash and cured for 28 days.

To investigate the change in mass of the medium gypsum soil in both N1 and N2 specimens, after the end of each cycle, the mass of each specimen was plotted in Figure 4.5. The N1 specimens for each curing period showed an increase in the mass in early cycles.

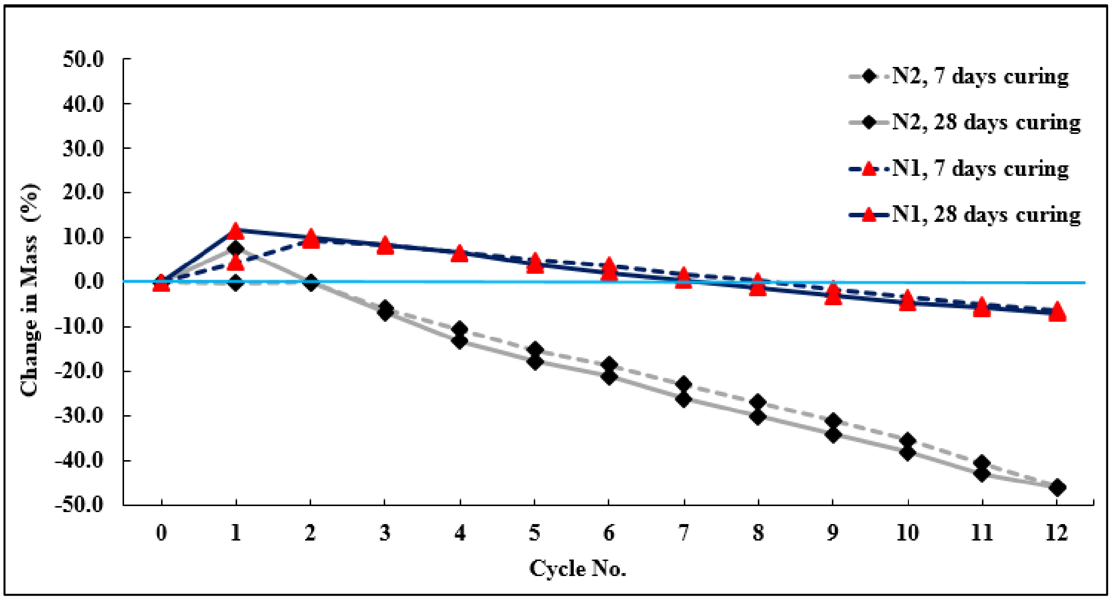


Figure 4.5 Change in mass for medium gypsum soil’s specimens treated with 30% activated fly ash and cured for 7, and 28 days.

This behavior occurred as the result of capsulated moisture inside the specimen, particularly when the drying temperature was not 100ºC, as recommended by the standard. In this case, a drying temperature of 35ºC was used for long time (about three days) to prevent the loss of gypsum due to the dehydration process.

However, after these increase in mass, a reduction occurred due to the increase in gypsum dissolution with cycles. N2 specimens showed the same trend at the beginning, followed by a significant loss. This trend was due to the brushing process after drying and the increase in gypsum dissolution during the wetting process of the cycles.

The total loss for most N2 specimens before reaching the end of the 12 cycles and the significant mass loss for the only two specimens that survived the 12 cycles was related to the aggressiveness of the mass loss (brushing operation) procedure of the ASTM D559/D559M – 15 standard. This standard states that the brushing operation that was used is the same operation in ASTM D560/D560M (Standard Test Method for Freezing and Thawing Compacted Soil-Cement Mixture). However, George and Davidson (1963) said *"Another severe test condition which does not simulate a field condition is the brushing weight loss of the specimen"* to describe the brushing process used in the ASTM D560.

4.3.3 Gypsum dissolution

4.3.3.1 The measured electrical conductivity for high and medium gypsum soils treated with activated fly ash

The electrical conductivity of the water bath after 5 hours of wetting for high and medium gypsum soils specimens are shown in Figures 4.6 and 4.7.

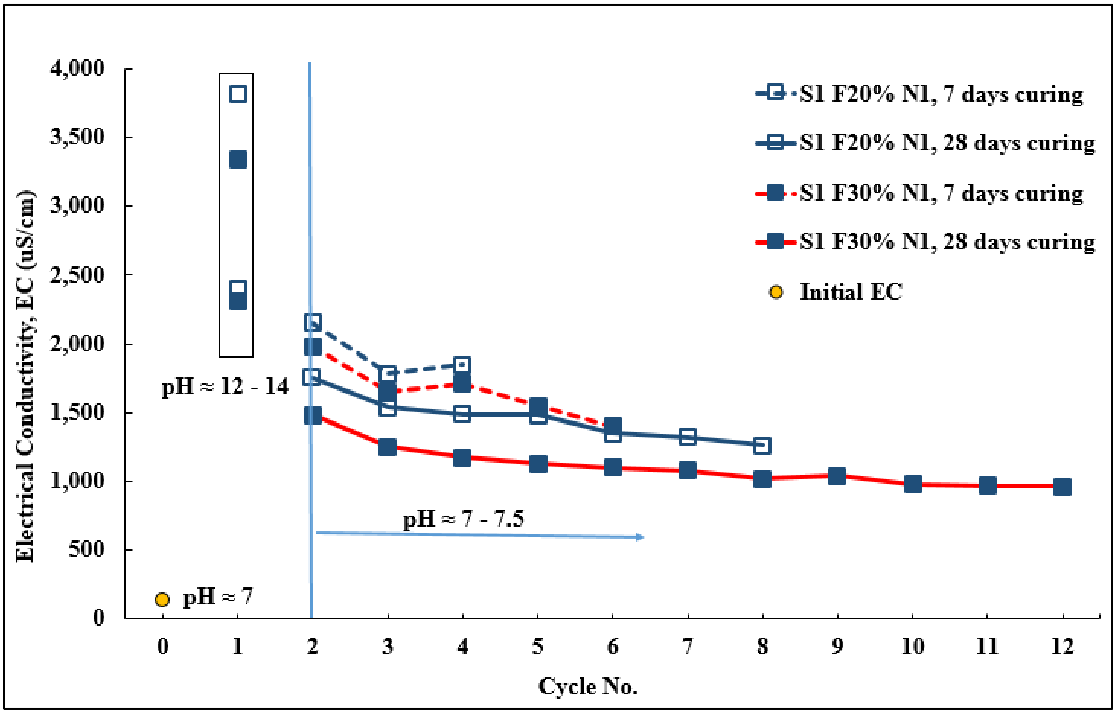


Figure 4.6 The measured electrical conductivity for high gypsum soil cured for 7, and 28 days.

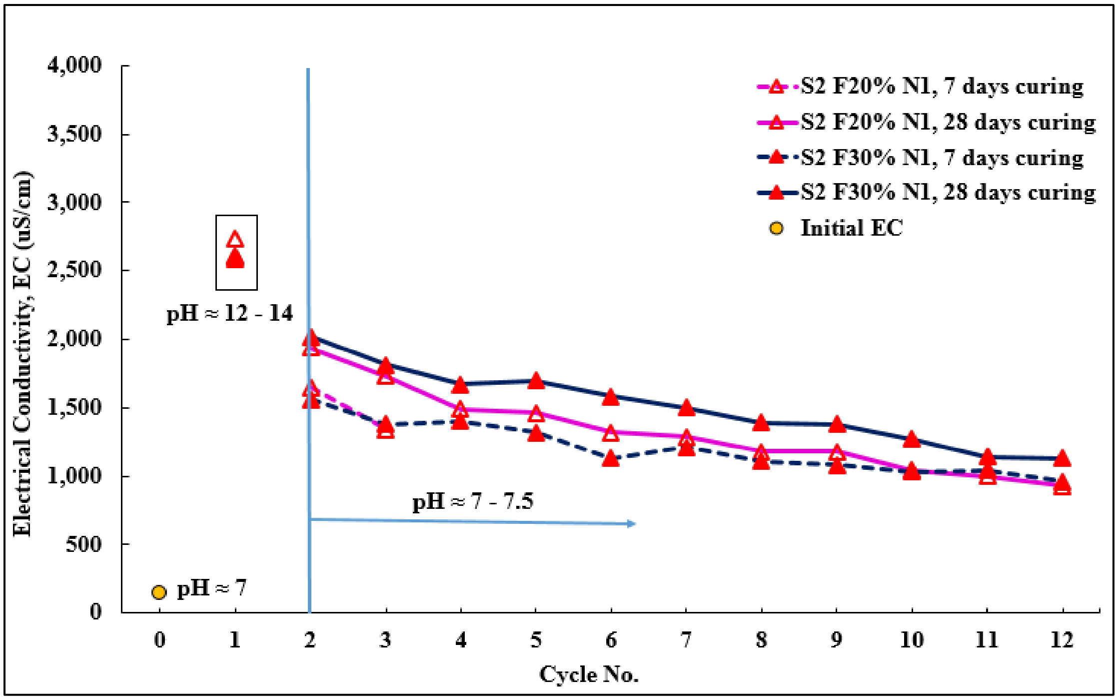


Figure 4.7 The measured electrical conductivity for medium gypsum soil cured for 7, and 28 days.

The first cycle in both soils show very high values, which gives an indication that these values may be a result of the non-reacting KOH or fly ash, particularly when the pH readings were found to be very high. (12-14). Therefore, separated tests for only fly ash specimens activated with fly ash and cured for 7 and 28 days were prepared to find the corresponding EC measurements.

The results of these tests showed very high electrical conductivity that increased with the specimen weight and had very high pH readings (between 12-14), even for a very small portion of the specimen’s weight.

This behavior indicates that the EC in the first cycle for both soils cured for 7 and 28 days is related to the dispersing of the non-reacting KOH and fly ash from the specimen. Therefore, the first cycles were eliminated.

In general, as shown in Figures 4.6 and 4.7, a reduction in the electrical conductivity was observed with the increase in cycles for both curing periods for both soils. This reduction means that less ions were found in the solution, which it is related to fewer materials being leached from the specimens with cycle increases.

The 28-day cured specimens for high gypsum soil showed a reduction in the electrical conductivity, compared to the 7-day cured specimens. This result was also supported by the findings of Alsafi (2017) and Jha and Sivapullaiah (2017), which show that more curing time will result in more activation and a more stable structure. However, reverse behavior was found between the 7 and 2-day cured specimens of medium gypsum soil, which may be related to the increase in voids between soil particles due to the ettringite formation, particularly the volume increased. The increase in voids allowed more water to move through the specimens, which increased gypsum dissolution.

4.3.3.2 The mass of gypsum

Equation 4.1 was used to estimate the amount of dissolved gypsum in each cycle from the EC measurements. To inveistigate the fate of the gypsum that was present in each specimen, a comparison was done in Table 4.4.

Table 4.4 Gypsum in Specimens Before, During, and After W-D Cycles.

Specimen	**Soil's gypsum content %[1]**	**Specimen gypsum content %[2]**	**No. of Cycles Survived**	**Total dissolved gypsum %[2] (from EC)**	**Remaining gypsum content % [3]**
High gypsum soil					
7 days curing					
S1 F20% N1	93	87.69	4	3.99	-------
S1 F30% N1	93	82.65	6	5.70	-------
28 days curing					
S1 F20% N1	93	83.09	8	6.41	70.31
S1 F30% N1	93	79.85	12	7.39	57.94
Medium gypsum soil					
7 days curing					
S2 F20% N1	31	27.39	3	1.75	-------
S2 F30% N1	31	25.65	12	7.67	5.34
28 days curing					
S2 F20% N1	31	27.47	12	8.42	14.25
S2 F30% N1	31	25.57	12	9.98	8.97

[1] Silica gel method, Nelson et al. 1978 was used to find the gypsum content.

[2] To the total original dry weight for each specimen.

[3] Remaining gypsum content in the specimen after W-D Cycles, the specimen crushed and three random samples were selected to find the gypsum content by the Silica gel method, Nelson et al. 1978, then the average was taken.

The gypsum content of each specimen was calcualted by dividing the amount of gypsum in the dry soil used to prepare the specimen by the original total dry weight of the specimen (the dry soil weight + activated fly ash weight). The total dissolived gypsum was estimated from the EC measurments by dividing the cumulative amount of dissolved gypsum from all the cycles that the specimen survived by the original total dry weight of the specimen.

However, the remaning gypsum content was found by using the silica gel method by crushing the specimen and taking the average of three representative samples. The estimate of the dissolved gypsum from the EC readings was based on an assumption that the ions in the water only came from the gypsum. This assumption represented a worst-case scenario because there are definitely others ions that leached into the water from the non-reactive KOH or from the fly ash.

Moreover, as has been mentioned in the mass loss section, the capsulated moisture inside the specimen due to the use of low drying temperature increased the mass in earlier cycles. Threfore, a comparision between these findings and the mass loss of N1 specimens cannot be done. From Table 4.4, it can be seen that for each specimen, an amount of gypsum is missing, which can be determined by comparing the original amount of gypsum in the specimen to the remaining amount after W-D cycles.

Although a small amount of gypsum was dissolved, but it was less than the difference between the original and the remaining amounts. This behavior indicated that some of the gypsum definity reacted with the activated fly ash, and a new chemical component resulted from this reaction, which was shown by Jha and Sivapullaiah (2017).

While the amount of dissolved gypsum in each cycle for each case showed a trend of reduction as the cycles increased, the percentage of cumulative dissolved gypsum showed an increase with cycles, as shown in Figures 4.8 and 4.9 for both high and medium gypsum soils.

The percentages of cumulative gypsum shown in these figures were calculated by dividing the cumulative amount of gypsum that was dissolved after all the cycles that the specimen survived by the original amount of gypsum in each specimen.

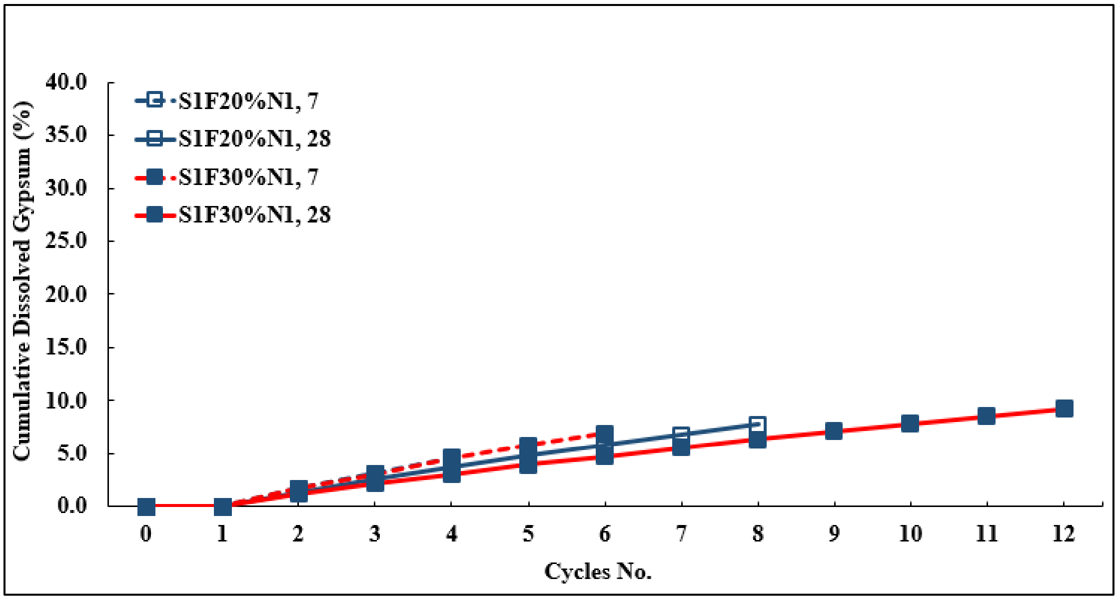


Figure 4.8 Effect of W-D cycles and curing time on cumulative gypsum dissolution/cycle for high gypsum soil.

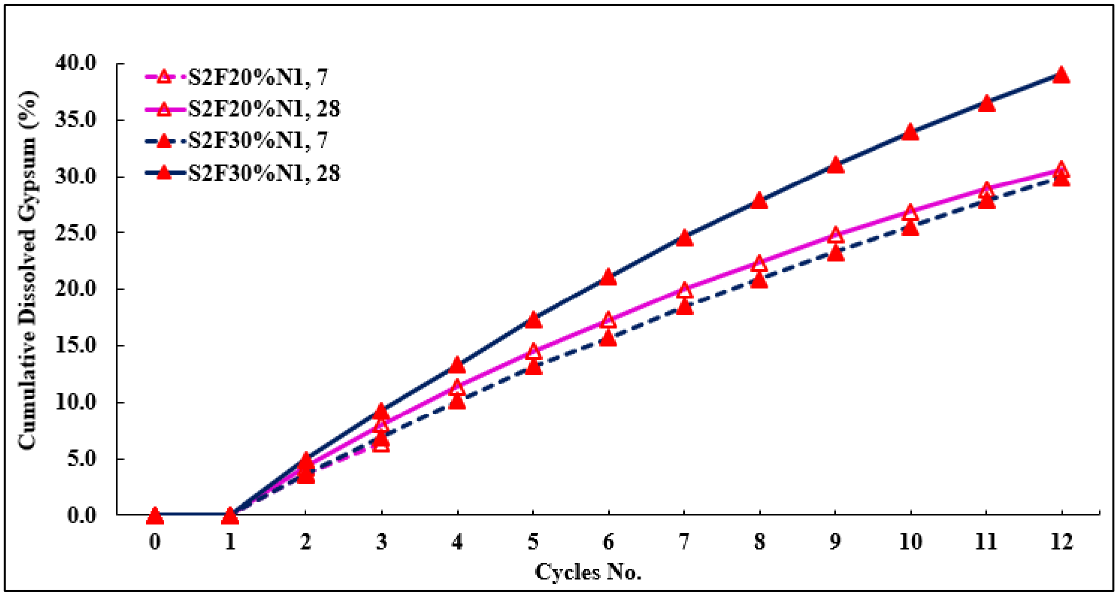


Figure 4.9 Effect of W-D cycles and curing time on cumulative gypsum dissolution/cycle for medium gypsum soil.

Figure 4.8 indicates that using higher activated fly ash amounts reduced the amount of dissolved gypsum for both curing periods. This estimate demonstrates that an improvement in the soil structure occurred with the increase of the stabilizer due to the formation of the ettringite and geopolymer gels.

The medium gypsum soil specimens also showed a different trend than the high gypsum soil, as shown in Figure 4.9. Higher activated fly ash amounts resulted in higher cumulative dissolved gypsum. Since gypsum dissolution was linked directly to the EC of the solution, this behavior was the same, as shown in Figure 4.7.

It can be described as follow: The formation of more ettringite due to the use of higher activated fly ash with higher curing periods resulted in the increase in the expansion of the specimen, which leads to the creation of more voids in the specimen structure, thus allowing for more water to infiltrate and dissolve more gypsum.

4.3.4 The unconfined compressive strength for W-D and control specimens

The unconfined compressive strength for both survived and control specimens are listed in Table 4.5. Some prepared specimens did not survive all the 12 cycles. Therefore, control specimens that matched the survived specimens were prepared and tested.

Table 4.5 Results of UCS and E for Survived and Control Specimens.

Specimen No.	**After W-D Cycles[1]**				**Control[2]**			
	7 days curing		**28 days curing**		**7 days curing**		**28 days curing**	
	UCS kPa	**E MPa**	**UCS kPa**	**E MPa**	**UCS kPa**	**E MPa**	**UCS kPa**	**E MPa**
High gypsum soil								
S1 F30% N1	---	--	973.36	104.94	501.54	62.20	1738.29	217.59
Medium gypsum soil								
S2 F20% N1	----	------	170.83	48.37	51.51	4.9	244.67	18.1
S2 F30% N1	635.29	34.16	371.48	27.87	33.42	1.11	187.04	16.5
Sterling Sand								
SS F30% N1	----	------	----	------	38.67	1.98	96.26	3.06

[1] One specimen for each.

[2] Average of three specimens.

As shown in the table, high gypsum soil control specimens showed higher strength, compared to the 28-day cured specimen that survived all 12 cycles, which means that the wetting-drying cycles impacted the strength of high gypsum treated soil.

On the other hand, medium gypsum soil specimens behaved differently, behaviors which was directly related to the amount of activated fly ash used in the treatment. For the 20% activated fly ash, the control specimen showed higher strength, whereas the 30% activated fly ash increased the strength of the survived specimens, as compared to the control specimens. In this case, the wetting-drying cycles appeared to extend the curing of the survived specimens.

However, for the same soil, the curing period has a different impact on the strength. Higher curing time resulted in lower strength for the surviving specimens. Conversely, the higher curing periods resulted in higher strength for the control specimens. Another finding can be inferred by comparing the results of the control sterling sand specimens with the high and medium gypsum soil specimens. This result directly corresponds with the findings that were discussed in the section of gypsum mass, which is the amount of gypsum that was not found in the specimens after the cycles.

As can be seen, the strength of the sterling sand specimens that were treated with 30% activated fly ash showed lower values, compared to the soils with gypsum that were treated with the same amount of activated fly ash. Higher gypsum content in the soil showed higher strength, which is directly linked to the suggestion that some of gypsum reacts with KOH-fly ash and creates new components that strengthen soil structure.

In addition, no sterling sand specimens that were treated with 30% activated fly ash survived the first hour of wetting during the first cycle. The findings of this study supported the results found in Jha and Sivapullaiah, (2017), where they mixed different dosages of gypsum with sandy soils and lime.

They found that the gypsum works as an activator for the pozzolanic reaction and the formation of ettringite. The available silica from gypsum also leads to the formation of C-S-H and the C-A-S-H gels. However, the formation of more ettringite with further curing leads to an opposite improvement (a reduction in the strength) due to the expansion and the deterioration of the soil structure.

4.4 Summary and conclusions

The objective of this chapter was to evaluate the behavior of high and medium sandy soils with gypsum that was treated with three different percentages of activated fly ash. The first part of this work was performed by using wetting – drying cycles on soil specimens that were cured for 7 and 28 days. The second part was performed by testing the unconfined compressive strength for the surviving specimens (i.e. specimens that finished all 12 cycles) and control specimens matched the treatment/curing of the surviving specimens.

1- Higher amounts of activated fly- ash are more suitable for use with granular soil with gypsum. Higher gypsum content requires higher amounts of activated fly ash to be used. Moreover, higher gypsum content results in higher UCS, as can be seen by comparing the 30% treated specimens.

   High gypsum soil that was treated with 30% and cured for 28 days had a UCS of approximately 973 kPa, but medium gypsum soil under the same condition had a UCS of approximately 371 kPa. Both specimens survived 12 cycles.

2- The use of the mass loss as an indication of the durability of granular soil may not be a good option because the ASTM D559/D559M – 15 standard has no specific statement to judge the durability of the specimen according to its mass loss.

3- The gain in the unconfined compressive strength is the result of several chemical reactions between gypsum-fly ash and minerals-KOH. These reactions occurred within the soil particles and changed the soil structure. Therefore, more investigation needs to be done to monitor the soil structure before/after curing and after the wetting-drying cycles.

4- Curing was found to be a key factor with the use of activated fly ash. Curing time affects the wetting resistance and soil structure for high gypsum soil. The 30% activated fly ash specimen had approximately 12% volume change when cured for 7 days, which was reduced to approximately 0% when cured for 28days. Moreover, the 7-day cured specimen did not survive the 12 cycles, but the specimen that was cured with 30% survived the 12 cycles and has a UCS of approximately 973 kPa. However, the curing effects on the medium gypsum soil were the opposite of the high gypsum soil.

The medium gypsum specimen treated with 30% and cured for 7 days had a volume change of 8%, which increased to approximately -17% (expansion zone). The 7-day cured specimen had a UCS of 635 kPa, which was reduced to approximately 371 kPa for a specimen that was cured for 28 days.

Chapter 5

The Effect of Static Water on the Behavior and Gypsum Dissolution for Gypseous and Gypsiferous Sandy Soils Treated with Asphalt Emulsion and Portland Cement

5.1 Introduction

The asphalt emulsion system consists of water (25-60%), emulsifier (0.1-2.5%), and asphalt binder (40-75%), along with other additives <2.5%. During the mixing process, the dispersed asphalt binder in water mixed with the soil, and the water helps mix these binders thoroughly with the soil particles. When the curing process starts, the water evaporates from the system, leaving a residue that consists of asphalt binders. These binders return to its solid stage in a process called coalescence and adhere to the surface of the soil particles.

This adhering mechanism improves the soil structure by adhering the particles to each other, which results in more condense and compact system. This system is also modified to be more water resistance, which results in high soil strength. These changes have been found with an increase in the percentage of asphalt emulsion in each soil. Taha et al. (2008) proved that an increase in the UCS occurred with an increase in the liquid asphalt percentage (cut-back RC-70) when they treated poorly graded sand with gypsum content that ranged between 40-50%.

However, there was an optimum liquid asphalt percentage (approximately 6%), after which the strength was reduced with increases in the liquid asphalt percentage. They also found that soaking the treated samples reduced the strength for all the liquid asphalt percentages, but in the case of soaking, the strength continued to increase with the increase in the binder percentage (the highest percentage that was evaluated was 8%).

Ahmed (2014) found that treating poorly graded silty sand, which had gypsum content of 49%, with asphalt emulsion increased the UCS; However, his results also showed that the optimum asphalt emulsion percentage of approximately 6%, and the strength was reduced beyond it.

5.2 Materials and methods

5.2.1 Soils

The soils used in this work were fine poorly graded sandy soils with gypsum (high gypsum soil (S1), medium gypsum soil (S2)) as described in previous chapters. To compare the results, a non-gypsum all-purpose silica sand soil (Sic) was also used in this work.

5.2.2 Asphalt emulsion

The asphalt emulsion that was used in this test is an anionic, slow-setting asphalt emulsion known as NTSS-1HM, produced by BLACLIDGE EMULSION, Inc., located in South Carolina, USA. It is often used as a tack coat in many applications, with a boiling point of 212ºF and a specific gravity, Gs of 1.03.

This type of emulsion was chosen to treat the natural soil because in many pavements’ applications, slow-setting types of emulsions are used with fine aggregates, as the surface area is large and requires time for uniform mixing.

5.2.3 Portland cement

The cement that was used in the work is Type I/II Portland cement.

5.2.4 Specimens preparation and curing

For asphalt emulsion treatment conducted in this study, three asphalt emulsion percentages were used (6%, 12%, and 18%), while one percentage (9%) was used for the cement treatment. In each case, two specimens were prepared: N1 and N2.

As described in the standard, during each cycle, the weight and volume of the N1 specimens were measured separately before and after wetting and drying. The weight of the N2 specimens were also recorded before and after wetting, as well as before and after applying strokes on each end and on the side of the specimen after drying.

2 in x 4 in. PVC mold was used to compact the specimens. The soil was mixed with the proposed asphalt emulsion by hand (mixing and kneading) for approximately 10 min. The mixture was compacted in the mold in three layers with a steel rod by applying 55 blows per layer. Then, the specimens were cured at 35ºC. The temperature was chosen to prevent gypsum dehydration during curing.

Curing time depends on the evaporation of the free moisture from the specimens. The weight of the specimen was taken each day until the difference between the last two weights was less than 0.5 g, which took approximately three days. The same procedure was used during the drying portion of each cycle. For specimens treated with Portland cement, 9% cement was used with 9% water content. The selection of the cement percentage was based on the soil classification.

Since both soils are type A-3, according to the ASHTTO classification, the range of the cement content that could have been used was between 7-11%, according to the soil-cement inspector's manual (Portland cement association, 2001).

The same compaction procedure was used to prepare the specimens, but in this case, the curing was different. In this case, the specimens were left inside the mold for 24 hrs. to solidify. Then, the specimens were removed from the molds, and the initial weights and volumes were recorded. All specimens were cured in sealed moist container for 7 days.

The moist container was filled halfway with water. The specimens were placed above the water level to prevent direct contact with water. Then the lid was placed, and the edges were sealed with duct tape. After curing, the specimens were removed from the container, and the weights and volumes were recorded. Table 5.1 identifies the shows specimens, along with the number of cycles that each specimen survived. In this table, S1 and S2 represented high and medium soils, and the % represented the asphalt emulsion percentage that was used to prepare the specimens.

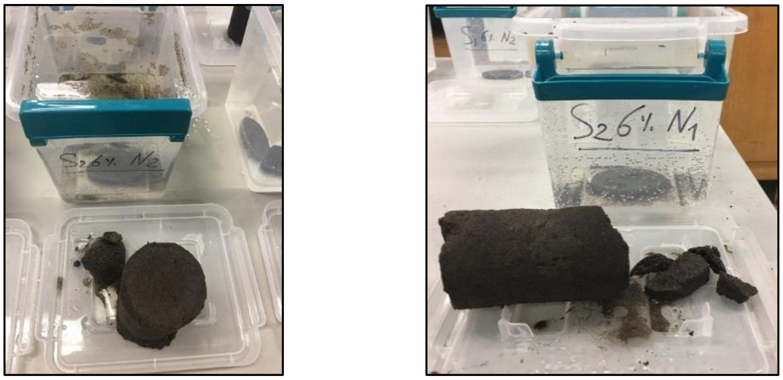
5.2.5 Wetting-drying test

The ASTM D559/D559M – 15 standard was used in the test. Modifications were made to the standard procedure, which are listed in Tables 5.2 and 5.3 for both treatments. During the wetting process, the electrical conductivity of the solution was measured. The electrical conductivity meter used in this work was purchased from HANNA instruments. It is a high range EC/TDS meter with a Model No. HI99301. The EC ranged between 0.00 to 20.00 mS/cm (0.00-20,000.00 µS/cm) with an EC resolution of 0.01 mS/cm (10 µS/cm).

Table 5.1 Soil specimen identification

Specimen ID	Cycles survived	Lost / Fell apart
Asphalt emulsion treatment		
High gypsum soil		
S1 6% N1	12	N/A
S1 6% N2	12	N/A
S1 12% N1	12	N/A
S1 12% N2	12	N/A
S1 18% N1	12	N/A
S1 18% N2	12	N/A
Medium gypsum soil		
S2 6% N1	W of the 9[1]	Before D of 9th
S2 6% N2	None[1]	W of the 1st
S2 12% N1	12	N/A
S2 12% N2	12	N/A
S2 18% N1	12	N/A
S2 18% N2	12	N/A
Silica sand soil (non-gypsum)		
Sic 6%N1	7	N/A
Sic 12%N1	7	N/A
Sic 18%N1	7	N/A
Portland cement treatment		
High gypsum soil		
S1 C9% N1	12	N/A
S1 C9% N2	12	N/A
Medium gypsum soil		
S2 C9% N1	12	N/A
S2 C9% N2	12	N/A

[1] Medium gypsum soil specimens treated with 6% asphalt emulsion (Figure 5.1); N2 failed in the first hour of wetting during the first cycle; N1 failed after finishing the wetting portion of the ninth cycle.



N2 lost during the 1st cycle. **N1 lost after W of the 9th cycle.**

Figure 5.1 Medium gypsum soil specimens treated with 6% asphalt emulsion.

Table 5.2 Deviations from ASTM D559/D559M – 15 for asphalt emulsion treatment.

Step	**Standard**	**Modification**	**Rationale**
Mold	4 X 4.5 in.	2 X 4 in.	Limited soil
Additive	Cement	Asphalt emulsion	Study objective
Curing	7 days, in moist room	Min. of 3 days, in 35º C oven	Constant weight, no gypsum dehydration
Vo of N1	Molding volume	After curing	Exact volume, neglect initial shrinkage
Wd in W/C, cys.	Original oven-dry mass	Dry weight after each cycle	Eliminate gypsum dissolution weight
Wetting	5 hrs. continuous	5 hrs., remove specimens each hr.	EC measurement
	No water changes	Water changes	No solution saturation
Drying	71º C, 42 hrs.	35º C, 3 days Min.	Constant weight
N2, strokes	4 each end, 20 side	One each end, 9 side	Smaller specimen
N2 Mass loss	B, standard table. 1	B=0	No water retains in spec.
	A= oven dry mass, 110º C	A= oven dry mass, 35º C	No gypsum dehydration

Table 5.3 Deviations from ASTM D559/D559M – 15 for Portland cement treatment.

Step	**Standard**	**Modification**	**Rationale**
Mold	4 X 4.5 in.	2 X 4 in.	Limited soil
Wd in W/C, cys.	Original oven-dry mass	Dry weight after each cycle	Eliminate gypsum dissolution weight
Wetting	5 hrs. continuous	5 hrs., remove specimens each hr.	EC measurement
	No water changes	Water changes	No solution saturation
Drying	71º C, 42 hrs.	35º C, 3 days Min.	Constant weight
N2, strokes	4 each end, 20 side	One each end, 9 side	Smaller specimen
N2 Mass loss	A= oven dry mass, 110º C	A= oven dry mass, 35º C	No gypsum dehydration

During the wetting process, the specimens were removed from the water each hour, and the time was stopped when the EC was measured. Afterwards, they were returned to the water until all five hours of the wetting process ended.

The EC measurements were used to estimate the amount of dissolved gypsum once the wetting process was finished by using linear regression equations for pure gypsum, high gypsum soil (S1), and medium gypsum soil (S2), which are listed in Chapter 3.

5.3 Results and discussion

5.3.1 Volume changes in N1 specimens during cycles

The volume changes for each N1 specimen during each cycle were calculated. The volume change during each cycle was measured twice, after wetting and after drying, by taking the difference between the current volume and the initial volume, which was the volume of the sample after curing. For cement treatment, the volume was taken exactly after removing the specimen from the mold. The volume measurement was done by taking the average of three diameters, and the three highest with 120° between each measurement.

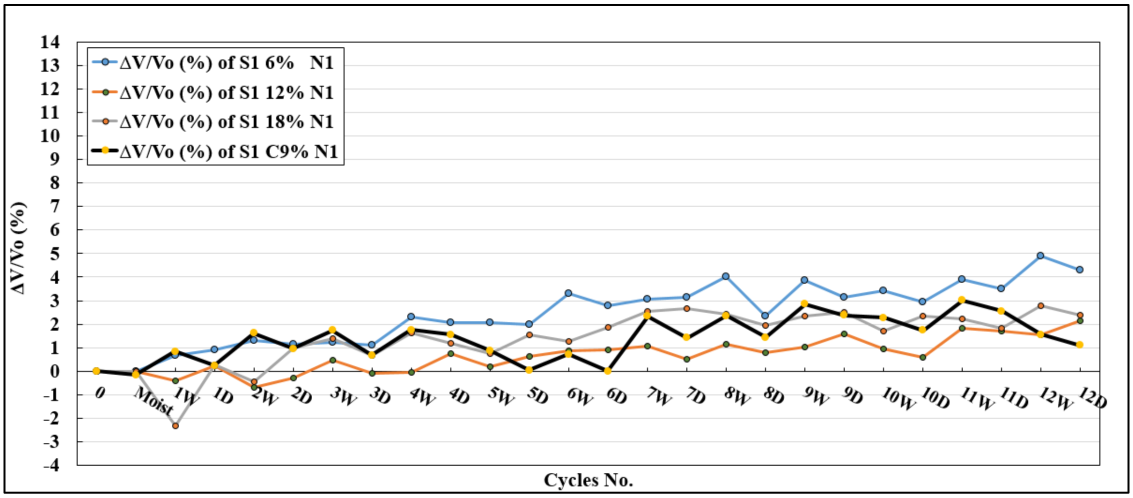
$$\frac{\Delta V}{Vo} = \frac{(V - V_o)}{V_o} \times 100 \qquad \text{(Eq. 5.1)}$$

$\frac{\Delta V}{V_o}$ = Volume change

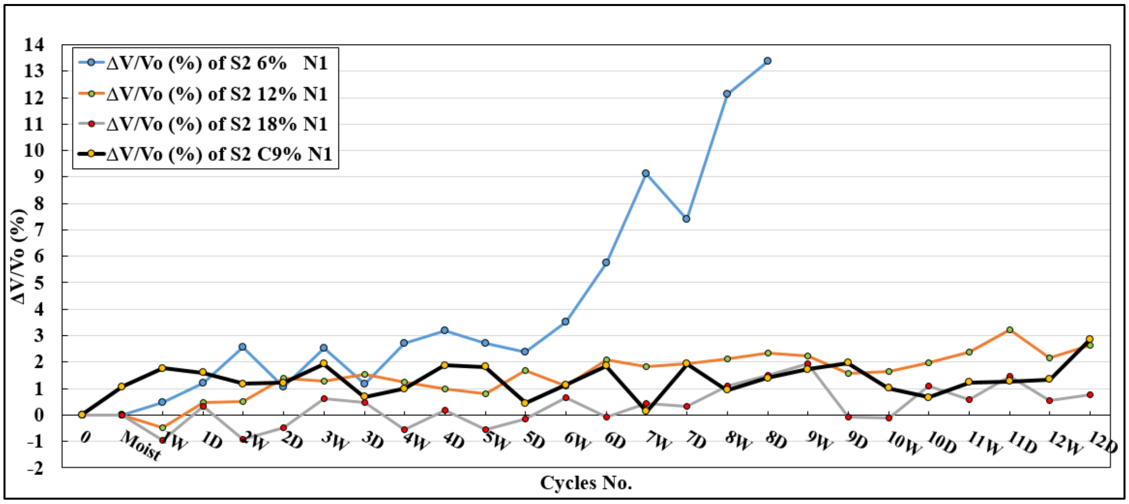
V = Volume of sample after wetting or drying.

V_o = Initial volume.

Figure 5.2 shows the volume change versus cycles for high and medium gypsum soils treated with asphalt emulsion and Portland cement.



High gypsum soil



Medium gypsum soil

Figure 5.2 Volume change vs. cycles for high and medium gypsum soils.

It shows that all specimens, despite the type of additive that was used for treatment, had a trend of overall slight shrinkage behavior with cycles, except for the medium gypsum soil specimen treated with 6% asphalt emulsion, which continue to shrink until it failed, prior to reaching the end of the 12 cycles.

12% asphalt emulsion provided the most stable volume for the specimen with high gypsum soil, whereas 18% asphalt emulsion treated specimen was more stable specimen for medium gypsum soil.

The use of asphalt emulsion improved the durability of soil with gypsum due to the improvement in the soils' cohesion when treated with asphalt emulsions, as shown by work conducted by Ahmed (2014) on poorly graded silty sand with gypsum content approximately 49% that was treated with asphalt emulsion.

The improvements of soils with gypsum that was treated with asphalt emulsion were related to the physical interaction and changes that occurred in the microstructure of the mixture due to the interlocking and interleaving process, which occurred between the calcium sulfate molecules (from gypsum) and the asphalt binders. No chemical interaction occurred between these two components, as was seen in the microstructure study that was done by Fan et al. (2019), where they examined the effects of different types of calcium sulfate whiskers on the performance of asphalt binder.

Cement treatment in both soils showed that 9% cement content provided volume change behavior that is closer to treating both soils with 18% asphalt emulsion, which is clear from both figures. However, 18% asphalt emulsion did not provide the lowest volume changes for high gypsum soil.

The initial improvement in the durability of the soil with gypsum treated with Portland cement is related to ettringite and formation of C-S-H, which was also found to cause deterioration due to the sulfate attack that occurred between the C-S-H and the sulfate ion (from gypsum), along with and the expansion related to the continued formation of ettringite upon wetting (Alsafi et al., 2017).

5.3.2 Water content changes in N1 specimens during cycles

The water content in each cycle was measured by taking the difference between the sample weights (after wetting and drying) and the original dry weight.

In this case, the original dry weight was the same as the drying weight in each cycle, as opposed to the initial dry weight after curing (which is a deviation from the standard). This approach was taken to minimize the errors related to weight loss due to gypsum dissolution during wetting.

$$ {}^{w}/_{C}\ \% = \frac{(W_w - W_d)}{W_d} \times 100 \qquad \text{(Eq. 5.2)} $$

${}^{w}/_{C}$ % = water content of sample.

W_w = Weight of sample after wetting.

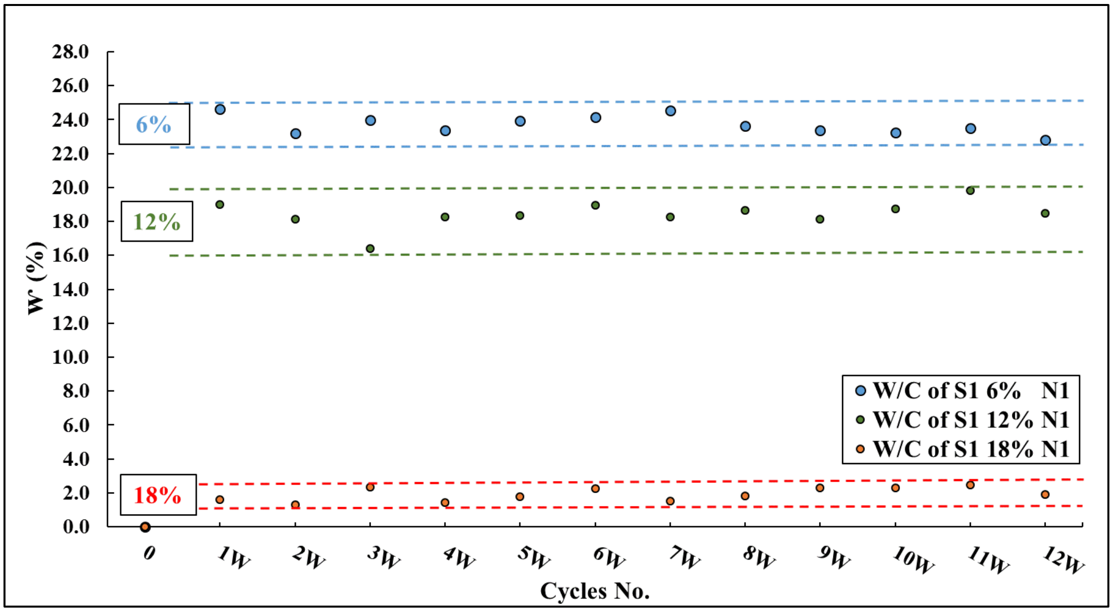
W_d = Weight of sample after drying.

Figure 5.3 shows the change in the water content for both soils treated with asphalt emulsion. The water content during drying was zero because the water content calculation used the drying weight as the initial dry wight. In both soils, the lowest water content was found in the specimens that were treated with 18% asphalt emulsion.

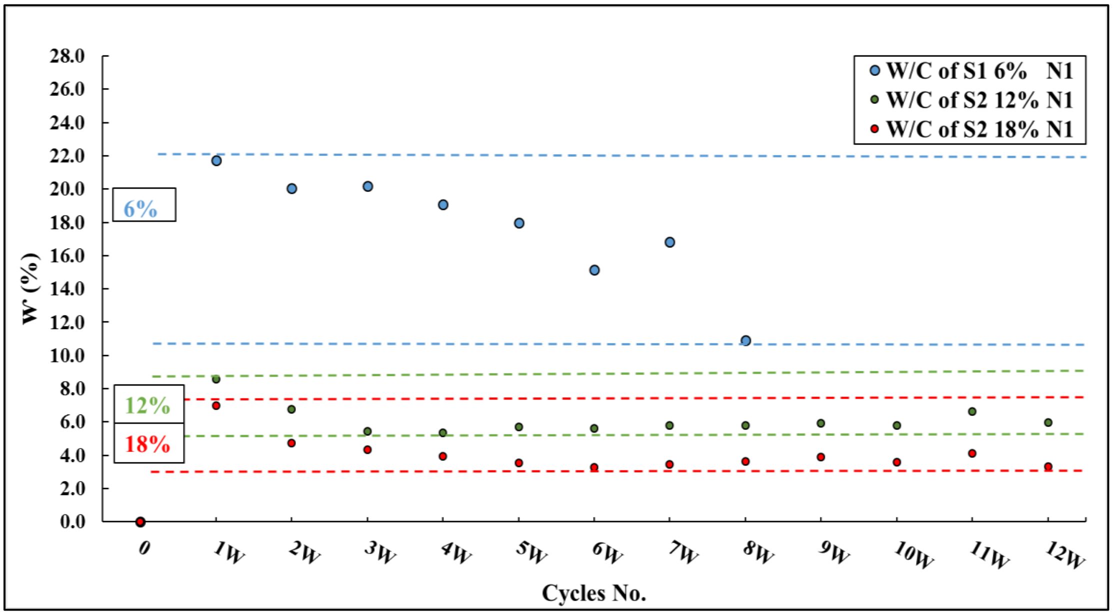
There was an interlock between the water content of the 12% and 18% asphalt emulsion treated specimens in medium gypsum soil. The water content was almost stable with cycles for each asphalt emulsion percentage, except for the medium gypsum specimen that was treated with 6% (failed specimen). A reduction trend was observed.

The water content results indicated that almost all specimens, with the exception of medium gypsum soil specimen that was treated with 6% asphalt emulsion, had very few changes in voids due to gypsum dissolution. This occurrence was considered to be an advantage for the use of asphalt emulsion as a treatment because any increase in water content would suggest that more gypsum was dissolved, and remained more voids in the system, as has shown by Aldaood et al. (2014). In their study, they found that an increase in water content occurred with increased cycles.

They examined the soil structure after wetting and found that more voids were created due to gypsum dissolution for low plasticity clay (CL) mixed with three different percentages of synthetic gypsum (5%, 15%, and 25%) and then treated with 3% lime.



High gypsum soil



Medium gypsum soil

Figure 5.3 Water content vs. cycles during wetting for high and medium gypsum soils.

5.3.3 Permeable porosity

To investigate how the asphalt emulsion and cement impacted the porosity of soils, the permeable porosity for each N1 sample was calculated by assuming that the volume of the pores was equal to volume of the water filling the pores and that all the pores are connected. The permeable porosity was calculated as follows:

$$n_p = \frac{V_p}{V} \qquad \text{(Eq. 5.3)}$$

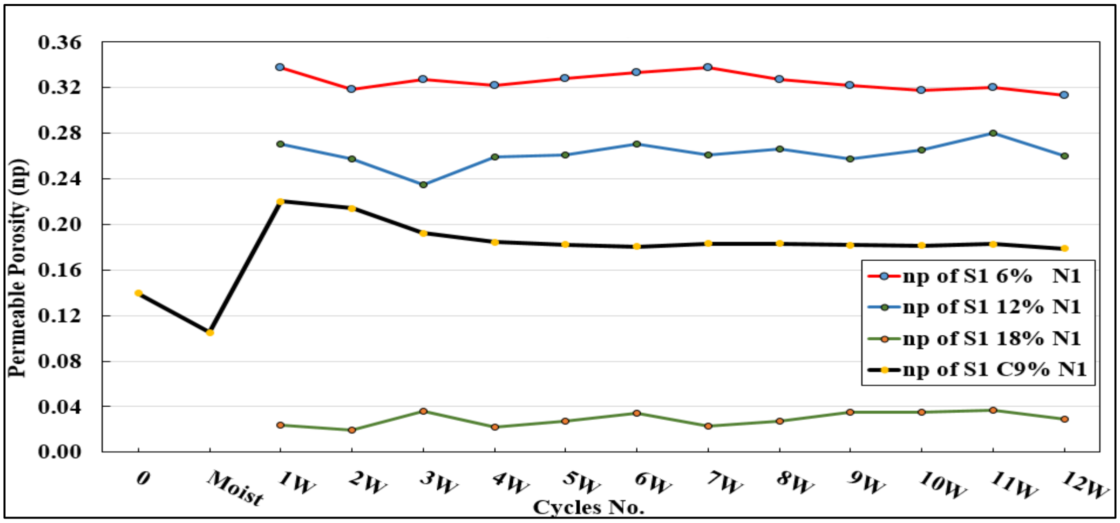
n_p = Permeable porosity.

V_p = Volume of pores = volume of water filling the pores ($V_w = W_w * \gamma_w$) after wetting.

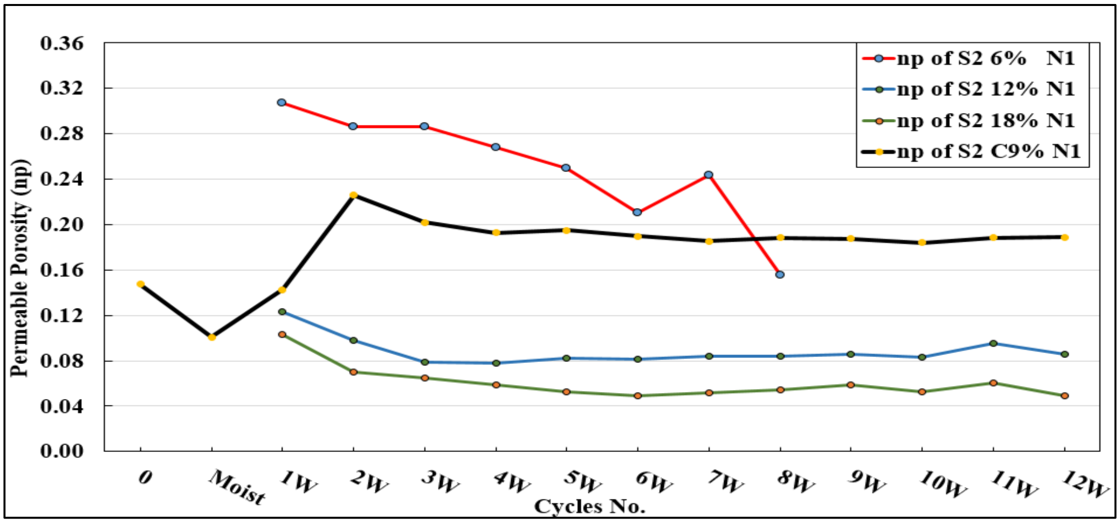
V = Sample volume after wetting.

Figure 5.4 shows the relationship between the permeable porosity for the wetting portion of each cycle for both soils. In both soils that were treated with asphalt emulsion, the permeable porosity was almost constant during all cycles, except the medium gypsum soil that was treated with 6% asphalt emulsion. The lowest permeable porosity was achieved by using the 18% asphalt emulsion, which was approximately 0.04 in high gypsum soil, and between 0.04-0.08 in medium gypsum soil.

Using cement as a treatment for both soils provided nearly the same permeable porosity in both soils (n_p about 0.2) during all cycles. These results support the idea that using the same cement content in both soils provided nearly the same soil structure, and as a result, the same pore space between particles, regardless of gypsum content, particularly when both soils are poorly graded fine sand. Nevertheless, treatment with cement is not preferable due to the deterioration that occurs due to the expected sulfate attack, which is the result of the presence of the sulfate ion from gypsum.



High gypsum soil



Medium gypsum soil

Figure 5.4 Permeable porosity in high and medium gypsum soils.

When comparing the results of the permeable porosity of cement treated samples with asphalt emulsion treated samples, it is evident that treating both soils with 18% asphalt emulsion provided the lowest permeable porosity, which is clear because using more asphalt emulsion will reduce the spaces between soil particles.

Although using cement did not prevent water from infiltrating between particles, in high gypsum soil, using cement as treatment provided a greater reduction in the permeable porosity than using 6% and 12% asphalt emulsion percentages, which is related to the fact that this soil has more gypsum to dissolve, thus it needs more emulsion to reduce the space between particles.

As has been observed, the porosity results emphasized that a reduction in gypsum dissolution reduction occurred by using asphalt emulsion as a treatment because the stability of the porosity showed fewer voids being created due to wetting. However, Aldaood et al. (2014) found an increase in porosity upon wetting due to the increase in gypsum dissolution for low plasticity clayey soils mixed with gypsum and treated with lime.

5.3.4 Soil mass loss

For each soil, the dry weights of the N2 specimens for each asphalt emulsion percentage and the 9% cement percentage were taken before starting the cycles and after finishing all twelve cycles. The mass loss percentage was calculated according to the ASTM D559/D559M – 15 standard with some deviations, as shown in Tables 5.2 and 5.3. The results are shown in Table 5.4. There is no value for S26%N2 because this specimen was lost during the first wetting cycle.

Table 5.4 Total mass loss of N2 specimens

Soil	**Asphalt emulsion**		**Portland cement**	
	Specimen	**Total mass loss %**	**Specimen**	**Total mass loss %**
Soil 1	S1 6% N2	37.33	S1 C9% N2	7.70
	S1 12% N2	6.51		
	S1 18% N2	3.85		
Soil 2	S2 6% N2	--------	S2 C9% N2	6.57
	S2 12% N2	15.86		
	S2 18% N2	5.23		

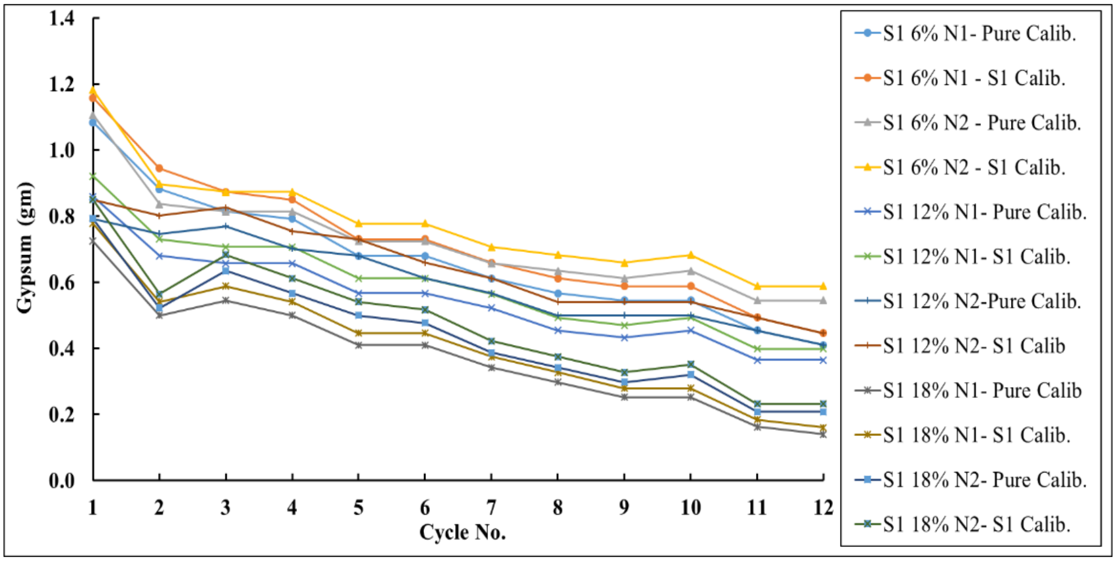
For mass loss, the same volume stability trend was shown by reducing the mass loss with an increase of asphalt emulsion. This trend occurred because any increase in the asphalt emulsion resulted in increased adhering between the soil particles and resulted in a denser soil structure, along with lowest gypsum dissolution. In cement treatment, the results showed that the high gypsum soil specimen lost more particles than the other soil, although the difference between the soils was not significant. However, these results suggest that the adherence between the cement and soil particles in medium gypsum soil was slightly higher.

By comparing the two types of treatments, it can be inferred that for high gypsum soil using 9% cement provided a mass loss that is similar to the use of the 12% asphalt emulsion, and for medium gypsum soil using 9% cement provided a mass loss similar to the use of the 18% asphalt emulsion.

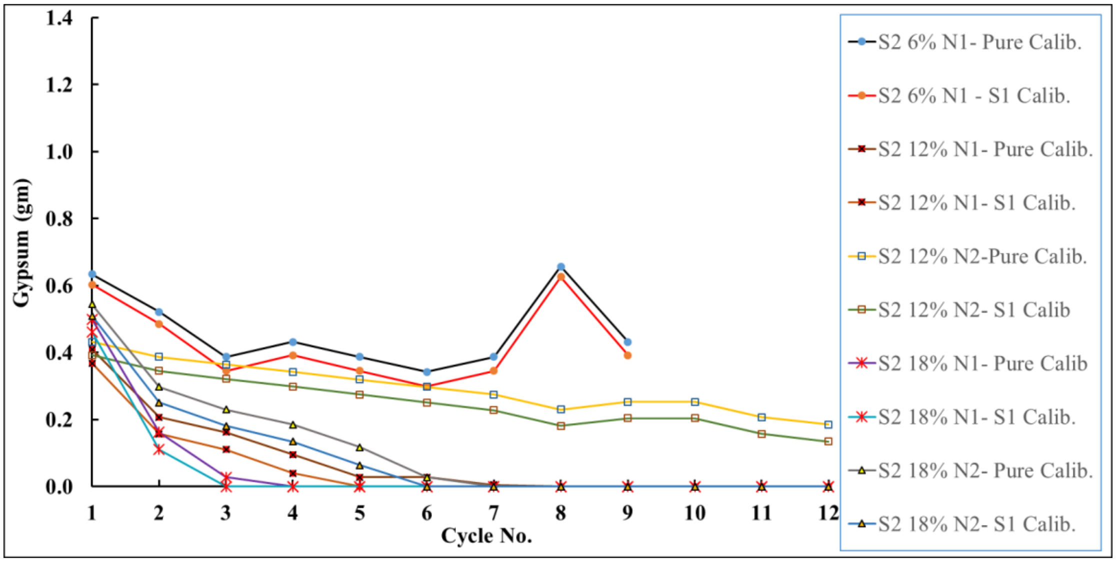
However, for both soils, treatment with 18% asphalt emulsion provided the lowest mass loss. This result may be related to the fact that using more emulsion results in more adherence between soil particles, less space between particles, and a more stable structure than using 9% cement. The results also suggest that 6% asphalt emulsion is not enough to prevent the mass loss in both soils. In general, the mass loss results showed that the use of asphalt emulsion as an additive to treat these two types of soil is a good approach to conserve the soil mass, compared to the use of activated fly ash, which was described in Chapter 4. The activated fly ash nerve prevents high gypsum soil specimens from falling apart (none of the N2 specimens survived the wetting-drying cycles), and the mass loss was approximately 45% for the both N2 medium gypsum soil specimens treated with 30% activated fly ash and cured for 7 and 28.

5.3.5 Gypsum dissolution estimation

The EC measurements was used to estimate the dissolved gypsum for each cycle. For specimens that were treated with asphalt emulsion, the results are shown in Figure 5.5, and the results for the cement treated specimens are shown in Figure 5.6. A reduction trend was observed in both soils. For asphalt treatment, higher asphalt emulsion percentage resulted in lower dissolved gypsum for each cycle.

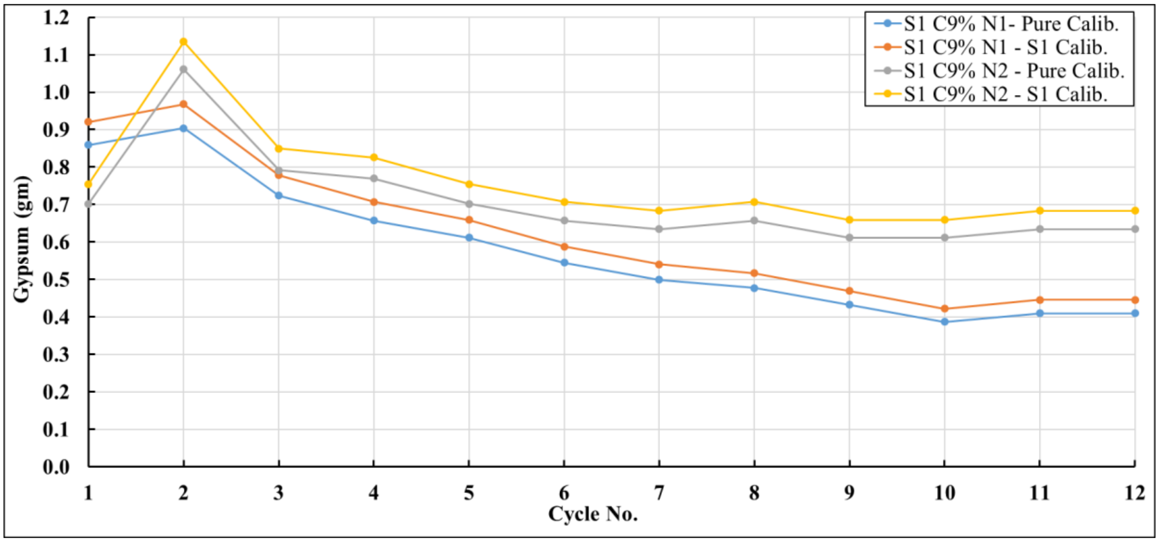


High gypsum soil

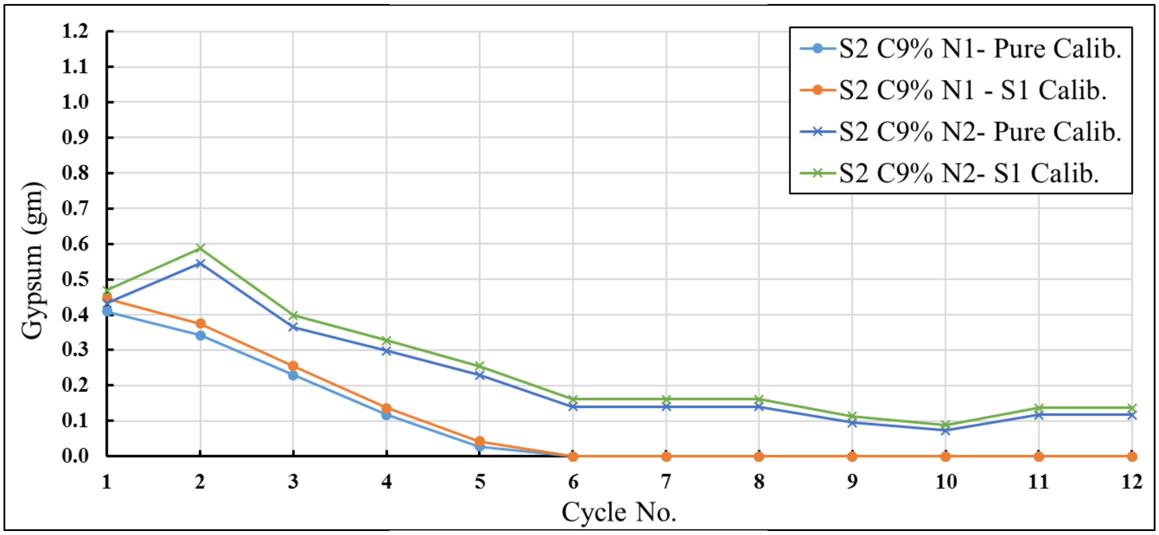


Medium gypsum soil

Figure 5.5 Gypsum dissolution / cycle for high and medium gypsum treated with asphalt emulsion soils.



High gypsum soil



Medium gypsum soil

Figure 5.6 Gypsum dissolution / cycle for high and medium gypsum treated with cement.

Medium gypsum soil specimens that were treated with 18% asphalt emulsion reached a state where no more gypsum dissolved (between the 6th and 7th cycle). For 9% cement treatment, it can be seen that the amount of dissolved gypsum was also reduced with cycle increases for both soils.

However, medium gypsum soil specimens showed lower values than high gypsum soil. Nevertheless, the N2 specimens in both soils showed higher values due to the brushing operation, which increased the amount of soil that was in direct contact with water.

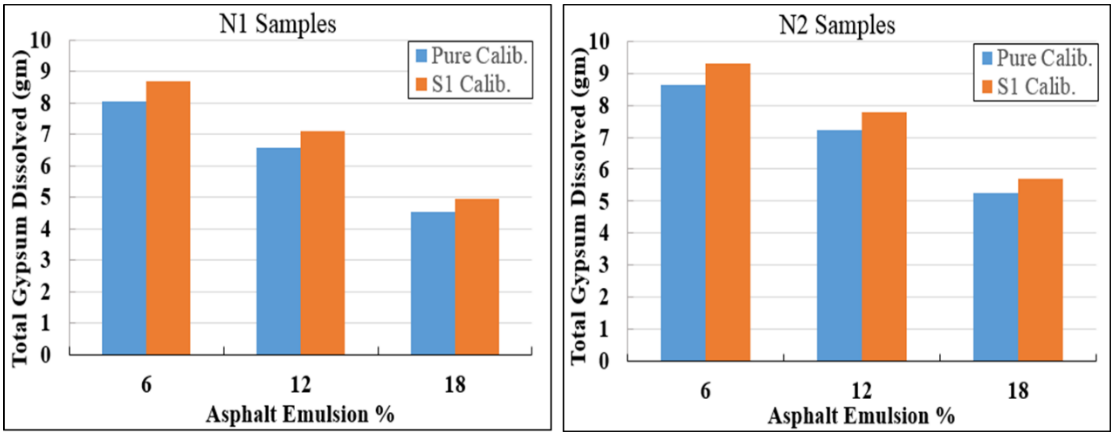
To be sure that the EC measurements in the water come only from the gypsum ions in the soil during the asphalt emulsion treatment and are not from the components of the asphalt emulsion, three specimens of non-gypsum silica sand treated with 6, 12, and 18% asphalt emulsion were prepared and cured according to the conditions of the gypsum soil specimens.

These specimens went through wetting-drying cycles with EC measurements as shown in Table 5.5. As can be observed, Cycles 1 and 2 had the highest EC measurements, which indicated that some chemicals from the emulsion leached into the water. After Cycle 3, the EC values were constant until Cycles 7. These small values after Cycle 3 were negligible, and the flocculation in the EC was noticed even in the pure tap water.

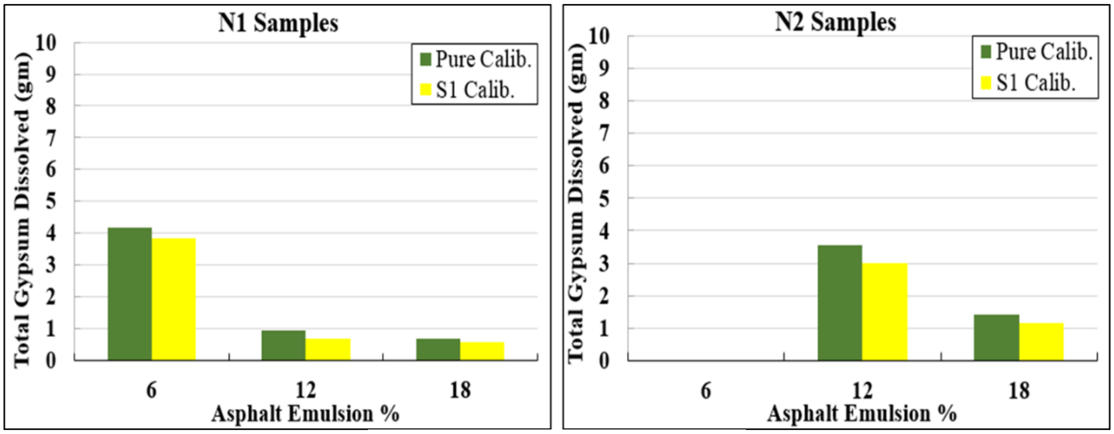
Table 5.5 The EC measurements in each cycle for silica sand specimens.

No.	**EC (µS/cm)**						
Cycle	**1**	**2**	**3**	**4**	**5**	**6**	**7**
Sic 6%N1	----	60	30	30	20	20	10
Sic 12%N1	50	30	10	10	10	10	10
Sic 18%N1	80	20	0	10	10	10	10

From the table above, it can be inferred that the asphalt emulsion had zero or relatively small effects on the EC reading in the gypsum soil specimens treated with the asphalt emulsion. The total dissolved gypsum dissolved in each soil for the N1 and N2 specimens treated with asphalt emulsion are shown in Figure 5.7.



High gypsum soil



Medium gypsum soil

Figure 5.7 Total gypsum dissolved in high and medium gypsum soils.

The results showed that more gypsum dissolved in the high gypsum soil specimens, compared to the medium specimens, but, in both soils, the N2 specimens lost more gypsum than the N1 specimens.

The highest gypsum dissolution occurred in the specimens treated with the 6% asphalt emulsion. Table 5.6 compares these results with the 9% cement treated N1 and N2 specimens. The maximum gypsum dissolution potential was estimated from the general gypsum dissolution rate (2.6 g/L) and the total amount of water used during the twelve cycles.

Table 5.6 Max. potential and measured gypsum dissolution in both soils.

Sample	**Water Vol. 1.8 L X 12**	**Total Gypsum Dissolution (gm)**		
		Max. potential from gypsum rate (2.6 gm/L)	**Measured from Pure Calib.**	**Measured from Soil Calib.**
High gypsum soil / asphalt emulsion treatment				
S1 6%N1	21.6	21.6 X 2.6 = 56.16	8.06	8.67
S1 6%N2	21.6	56.16	8.64	9.29
S1 12%N1	21.6	56.16	6.58	7.10
S1 12%N2	21.6	56.16	7.23	7.79
S1 18%N1	21.6	56.16	4.53	4.94
S1 18%N2	21.6	56.16	5.25	5.70
High gypsum soil / cement treatment				
S1 C9% N1	21.6	56.16	6.92	7.46
S1 C9% N2	21.6	56.16	8.47	9.10
Medium gypsum soil / asphalt emulsion treatment				
S2 6%N1	21.6	21.6 X 2.6 = 56.16	4.18	3.83
S2 12%N1	21.6	56.16	0.93	0.67
S2 12%N2	21.6	56.16	3.54	2.99
S2 18%N1	21.6	56.16	0.69	0.57
S2 18%N2	21.6	56.16	1.40	1.13
Medium gypsum soil / cement treatment				
S2 C9% N1	21.6	56.16	1.13	1.26
S2 C9% N2	21.6	56.16	2.69	2.99

From the table, it was determined that 9% cement did not prevent gypsum from dissolving. Although, the reduction was much less than the maximum potential dissolution of gypsum, it was not as low as the 18% asphalt emulsion in both N1 and N2 specimens for both soils.

5.4 Summary and conclusions

In this chapter, the effect of static water was studied to evaluate its influence on soil durability (volume, mass) and gypsum dissolution by using wetting-drying cycles on sandy soils with gypsum treated with different additives.

The first portion of this work was performed by using wetting – drying cycles on soil specimens treated with three different asphalt emulsion percentages (6%, 12%, and 18%).

The second portion was a control test that was conducted by using the same wetting-drying cycles on samples that were treated with 9% Portland cement because the standard that was used in this work was originally designed to evaluate the durability of compacted soil-cement mixtures. The following conclusions were found from this study:

1- Using 6% asphalt emulsion was not enough to stabilize the volume changes or to mitigate mass loss. N1 and N2 medium gypsum soil treated with 6% asphalt emulsion did not survive the twelve cycles. The N2 specimen was lost during the wetting portion of the first cycle, and the N1 specimen was lost during the wetting portion of the ninth cycle. The N1 specimen of high gypsum soil treated with 6% asphalt emulsion showed the highest changes in volume and water content, compared to other asphalt emulsion percentages. The N2 specimen with the same percentage showed the highest mass loss.
2- The results also showed that the medium gypsum soil with gypsum content of 31% shrank less than Soil 1, which had gypsum content of approximately 93%. This behavior was related to gypsum dissolution because it had the lowest gypsum content. In both soils, shrinkage behavior generally increased with cycles, whereas the water content showed constant behavior with cycles, which was seen in the 9% cement treated specimens.
3- The shrinkage behavior and the water content of the N1 specimens decreased with an increase in the asphalt emulsion percentage.

In high gypsum soil, 12% asphalt emulsion specimen showed the lowest volume changes, and in medium gypsum soil, 18% asphalt emulsion showed the lowest volume changes. For water content in both soils, 18% specimens showed the lowest changes in water content.

4- Using asphalt emulsion greater than 12% is a good approach to treat these soils because it reduced the permeable porosity to very low values, which resulted in volume stability and the lowest mass loss.

5- The 9% cement treatment also stabilized the volume in both soils, such that the volume was similar to the higher percentage of asphalt emulsion (12 and 18%). However, it did not close the voids as the asphalt emulsion did, which is clear from the permeable porosity results.

6- Although the cement treatment showed good improvement, it is not preferable due to the sulfate attack that would be initiated upon wetting as a result of the reaction between the cement components and the sulfate ions from gypsum in the soil in the presence of the water.

Chapter 6

The Effect of Moving Water on the permeability and the Gypsum Dissolution for Gypseous and Gypsiferous Sandy Soils Treated with Asphalt Emulsion

6.1 Materials and methods

6.1.1 Soils

The same medium and high gypsum soils described earlier are used in this work

6.1.2 Asphalt emulsion

The same asphalt emulsion described in Chapter 5 was used to prepare the treated samples. Two percentages of asphalt emulsion, 6% and 18% were used in the treatment, depending on wetting-drying results, which represents a static water conditions.

6.1.3 Methods

Two standard methods were used in this work. The first standard was the constant head procedure listed in ASTM D2434-68 (2000), which used a permeameter cell with a diameter of 2.5 in. and a sample height of 6 in., along with a head of 17.8 cm and an initial gradient of 1.18.

This procedure was used to measure the coefficient of permeability for the untreated soil samples and for one set of treated samples, with leachate collection. In this case, the treated samples were mixed with 6% asphalt emulsion, then cured at 35ºC and compacted inside the cell.

The dry unit weight that was used to prepare the sample was similar to the dry unit weight of the 6% treated samples, which were used in the wetting-drying study in Chapter 5. Compaction was conducted by dividing the soil weight into three batches and they were compacted in three layers inside the cell with a steel rod.

The second method is a combination of two procedures from ASTM D5084-16a and ASTM D2434-68 (2000). Both soil samples were treated with 18% asphalt emulsion. In this case, the sample height and diameter were the same as the previous method; however, the sample was compacted in a split PVC mold and then cured at 35ºC.

After the sample reached a constant weight, it was placed in 2.5 in. membrane and installed inside a flexible wall cell for testing. The procedure consists of the following: A flexible wall cell is used to measure the coefficient of permeability for the two soils by setting up the sample in the cell according to the procedure described in ASTM D5084, applying confining pressure of no more than 5 psi to hold the sample without falling, and attaching the membrane to the sample to prevent any water leaks.

The sample was saturated by applying vacuum pressure from the top nozzle, closing the bottom nozzle by applying a vacuum pressure of 20 Hg for 15 min., and then opening the bottom nozzle to allow water from a container to slowly flow through the sample from the bottom to the top slowly until water can be seen in the hose (according to ASTM D2434).

After saturation, the constant head test began by following the same procedure outlined in ASTM D2434, which has been used to test untreated soil samples by applying a head of 17.8 cm and then measuring the coefficient of permeability (k) directly after opening the nozzles with leachate collection.

During both tests, the pore volume of each sample was calculated. Then, after starting the test, an initial k value was measured by taking the average of three readings. Afterwards, the test was repeated after one pore volume passed through the sample. It is important the mention that the water flow remained open from the beginning of the test until the decision was made to terminate the test.

The leachate was collected from the beginning of the test, and for each k measurement, the EC conductivity of the leachate volume collected was recorded. This value was used to estimate the dissolved gypsum, as described previously.

6.2 Results and discussion

6.2.1 Untreated soils tests

To investigate the relationship between the coefficient of permeability, k, and the leachate volume, they were plotted on Figure 6.1 for untreated high and medium gypsum soil samples. High gypsum soil has the highest k, and more water flowed through it, compared to medium soil within the same time. The trend shows that k decreased in both soils with water flowing. rate.

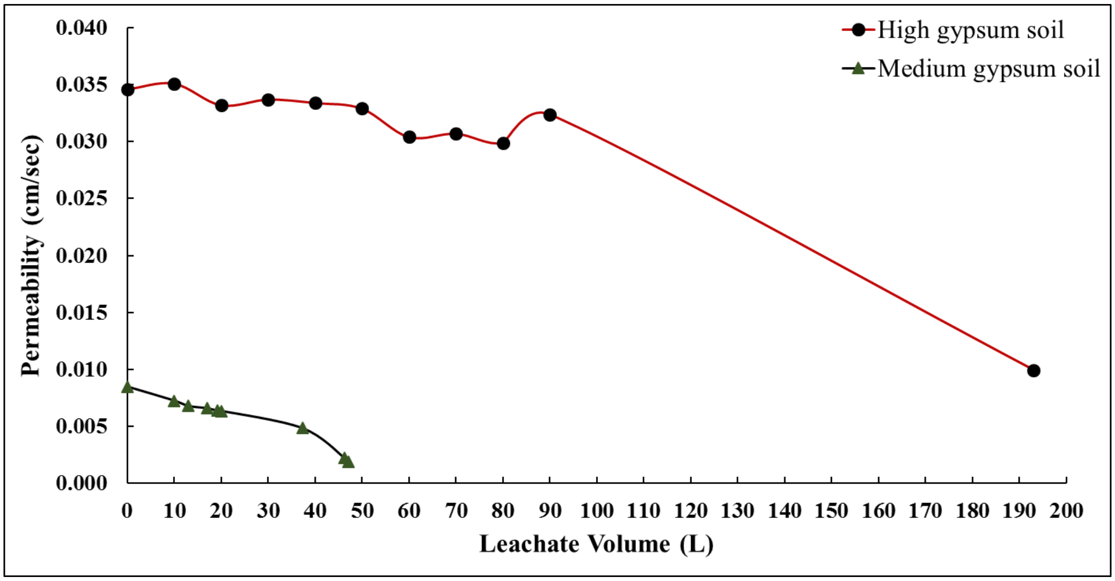


Figure 6.1 Coefficient of permeability for untreated high and medium gypsum.

The reduction of k is related to the change in the soil structure, which is related to the movement of particles to close the voids, reduce the micro tubes in the soil, and reduce the flow

Kuttah and Sato (2015) linked the permeability in soil with gypsum to two factors: gypsum content in soil and the size of gypsum particles. The permeability of soil with gypsum increased with an increase in the gypsum content, which only occurred when the gypsum particles were larger than the soil particles due to the dissolution of gypsum. while the permeability of the soil with gypsum decreased with an increase in gypsum content when the gypsum particles were smaller than the soil particles, in this case, when gypsum particles began to dissolve and move with the water, it closed the water paths and reduced the overall soil permeability.

The EC measurements show that these values remained constant at approximately 2200 µS/cm for high gypsum soil and 2000 µS/cm for medium gypsum soil for the entire test, as shown in Figure 6.2. This behavior indicates that the gypsum started to dissolve from the beginning until the end of the test at same rate.

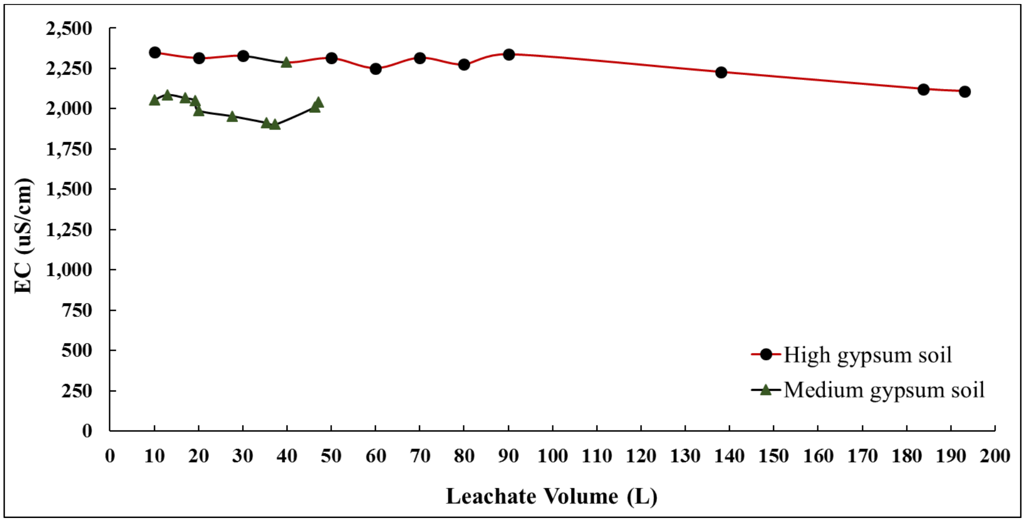


Figure 6.2 EC measurements for untreated high and medium gypsum soils.

The EC readings in both soils were used to estimate gypsum dissolution and were plotted against the leachate volume in Figure 6.3 for high and medium gypsum soil. This figure shows the cumulative dissolution. The dissolution of gypsum was constant with leachate volume and similar to the general rate of dissolution, which is 2.6 g/L.

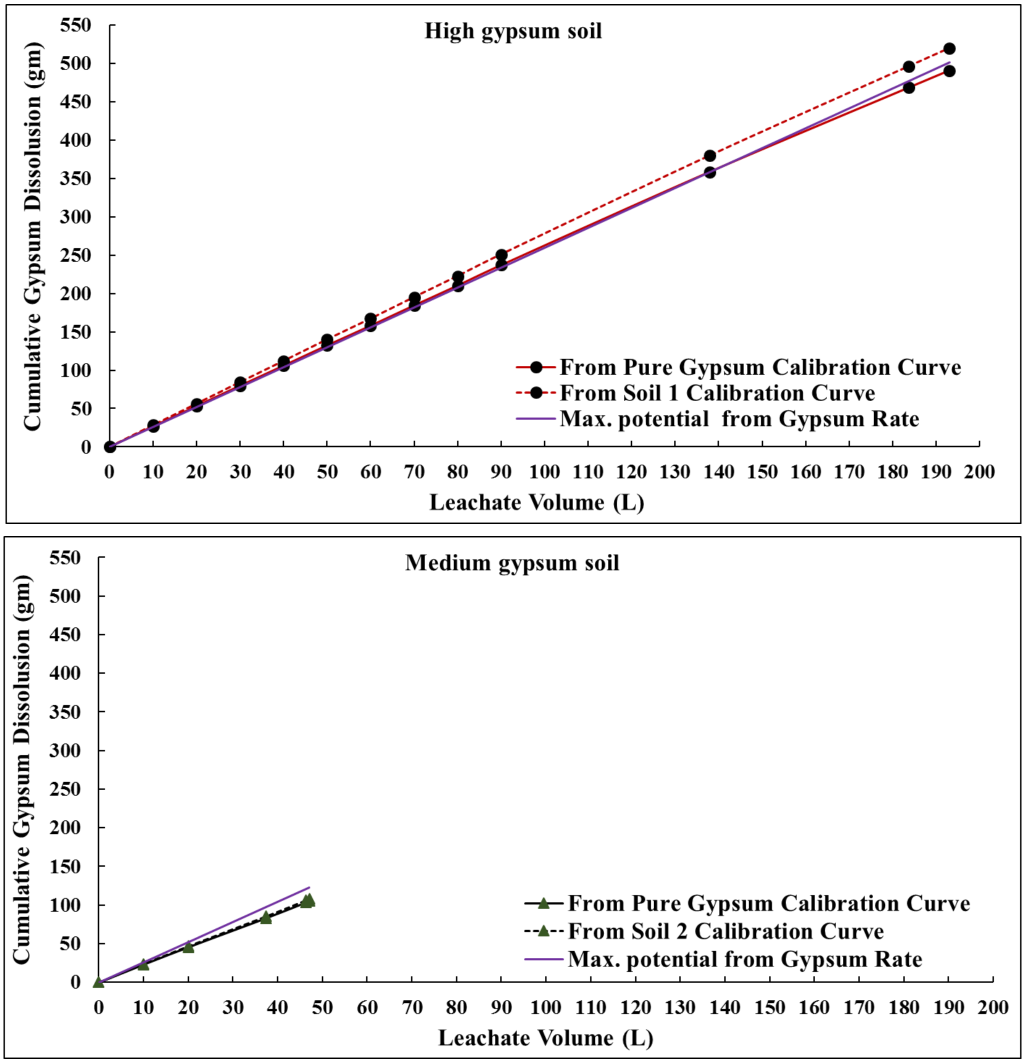


Figure 6.3 Gypsum dissolution for untreated high and medium gypsum soils.

The lowest total amount of dissolved gypsum in the medium gypsum sample was not related to the lower amount of gypsum, when compared to the high gypsum sample, but was related to the fact that less water moved through the sample because this soil had lower permeability.

The mass loss was very clear when comparing the samples before and after testing. Figure 6.4 shows the mass loss in both samples, which occurred in both soils. However, high gypsum soil lost more mass due to its higher coefficient of permeability and higher amount of gypsum. This mass loss was only related to gypsum dissolution. In other words, no particles were allowed to seep out with the leaching water.

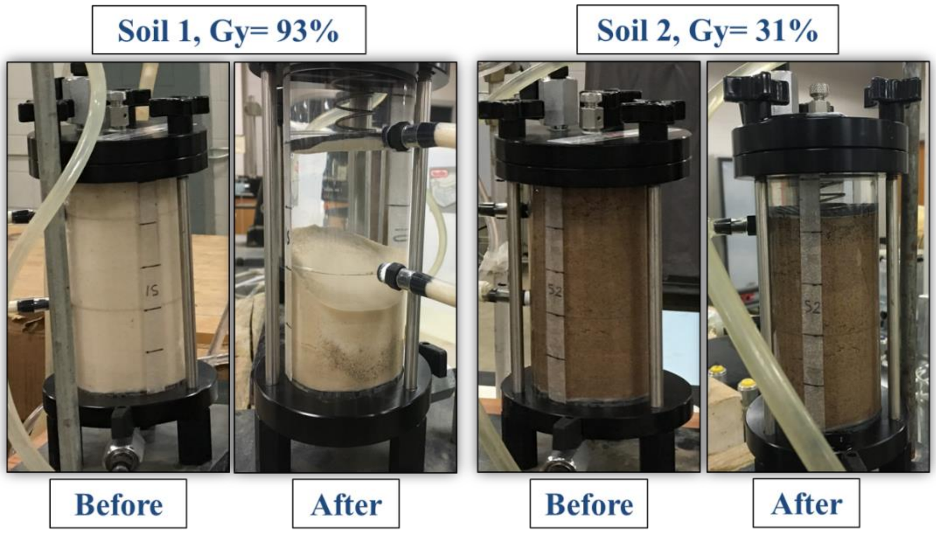


Figure 6.4 Mass loss due to gypsum dissolution for untreated high and medium gypsum soils.

6.2.1.1 Modeling the relation between the permeability and gypsum dissolution

The permeability for untreated soils indicated a strong relationship between the dissolution of soluble solids (i.e., gypsum) and soil coefficient of permeability. Many empirical formulas were used to predict the coefficient of permeability for different types of soil. However, limited prediction formulas are available that connect the soil coefficient of permeability with the dissolution rate of different soluble minerals.

6.2.1.1.1 Baena and Toledo, 2014 model

Baena and Toledo (2014) used the Kozeny–Carman (Kozeny 1927; Carman 1956) equation (Eq. 6.1) to produce a model, which they verified it experimentally, to investigate the seepage, which will occur under the foundations due to mineral dissolution when subjected to the intergranular flow of water.

$$k = \frac{g\rho}{v} \frac{1}{5} \left(\frac{V_g}{S}\right)^2 \frac{n^3}{(1-n)^2} \qquad \text{(Eq. 6.1)}$$

k = Coefficient of permeability.

g = Gravitational acceleration constant.

ρ = Fluid density.

$\boldsymbol{v}$ = Fluid viscosity.

Vg = Volume of grain.

S = Surface area of grain.

n = Porosity.

To estimate the initial coefficient of permeability (k_o), they used Eq. 6.1 directly with the initial porosity (n_o), as follows:

$$k_o = \frac{g\rho}{v} \frac{1}{5} \left(\frac{V_g}{S}\right)^2 \frac{n_o^3}{(1-n_o)^2} \qquad \text{(Eq. 6.2)}$$

To estimate the coefficient of permeability at a specific time after the dissolution of the soluble minerals began, the following terms were used:

V_t = soil volume

$V_t = V_h + V_s + V_{in}$

V_h = volume of interconnected pore holes

V_s = volume of soluble particles

V_{in} = volume of insoluble particles

- The effective porosity before dissolution $n_o = V_h / V_t$

- They assumed that when dissolution occurs, the effective porosity increases.

The post-dissolution effective porosity (n)

$$n = \frac{V_h + V_s}{V_t} = n_0 + \phi$$

$Ø = V_s / V_t$ (with Ø being the percentage in volume of soluble material)

The coefficient of permeability after dissolution (k_{td}) will be found by using the same equation (Eq. 6.1) but by plugging in the post-dissolution effective porosity (n).

$$k_{td} = \frac{g\rho}{v}\,\frac{1}{5}\left(\frac{V_g}{S}\right)^2 \frac{n^3}{(1-n)^2} \qquad \text{(Eq. 6.3)}$$

k_{td} = The coefficient of permeability after dissolution.

In their assumption, all the factors, such as the liquid density and viscosity, along with the ratio between the volume of the grain and its surface area, are constant. Then, the coefficient of permeability after dissolution (k_{td}) will be determined by dividing Eq. 6.3 by Eq. 6.2.

$$k_{td} = k_o \frac{(1-n_o)^2 n^3}{n_o^3 (1-n)^2}$$

$$k_{td} = k_o \frac{(1-n_o)^2 (n_o + Ø)^3}{n_o^3 (1-n_o-Ø)^2}$$ **Baena and Toledo, 2014 model** (Eq. 6.4)

They proved the model experimentally by performing two seepage tests on Hostun sand. The first test was conducted by mixing the sand with high soluble mineral (sodium bicarbonate with solubility rate of about 106 g/L). The second test (long test) was conducted by mixing the sand with natural gypsum, which has a purity of approximately 75% (solubility rate of about 2.4 g/L).

6.2.1.1.2 The influence of Ø on k prediction

In this study, prior to using the Baena and Toledo 2014 model, the model was tested within a range of initial porosity (n_o) to find the relationship between (k_{td} / k_o) and the values of Ø, as shown in Figure 6.5.

n_o values were selected within the range of 0.3 – 0.46 to match the range of granular soil found in Foundation Engineering, 2nd Edition (Peck, Hanson, and Thornburn, 1974). Ø values were selected to start from zero (no dissolution occurs), to the highest value of Ø =0.5 to match the highest value of n_o.

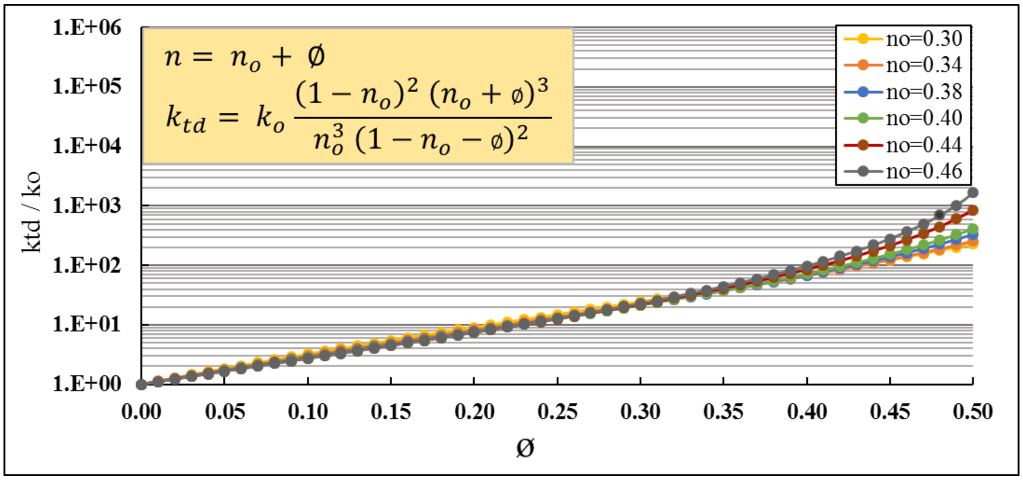


Figure 6.5 Baena and Toledo, 2014 model for a specific range of n_o and Ø.

The range of the k_{td} / k_o was selected to start from a value of 1 (when there are no changes in n and Ø, such that k_{td} = k_o). The highest value of 1000 was chosen by assuming that the highest value of k_{td} would be 1000 times the k_o value.

The relationship shows that k_{td} / k_o (simply k_{td}) increased with an increase in Ø for all the n_o values. The behavior of the soils with different initial porosities is similar until the value of Ø is approximately 0.4. Beyond this value, it can be seen that for the same value of Ø, the permeability value increases with higher value of n_o.

This model has a limit, which occurs when the value of n_o + Ø = 1. This value is not feasible because in this instance, n =1 ($n = V_v / V_t$). In other words, all the soil volume turns into voids (all the solids have been dissolved).

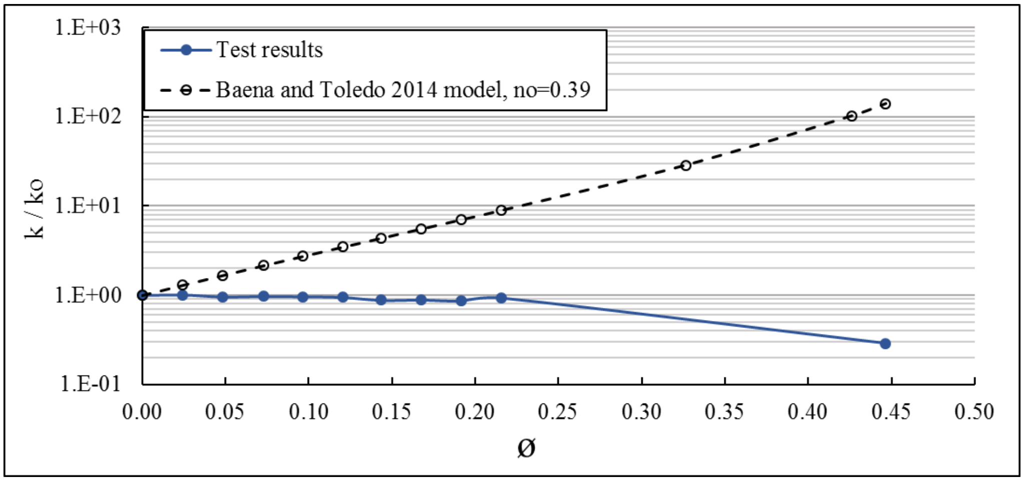
6.2.1.1.3 Using Baena and Toledo, 2014 model for untreated soils results

In this section, the Baena and Toledo (2014) model was used to predict the relationship between the coefficient of permeability and the volume of dissolved gypsum, which was represented by the value of Ø for high and medium gypsum soils.

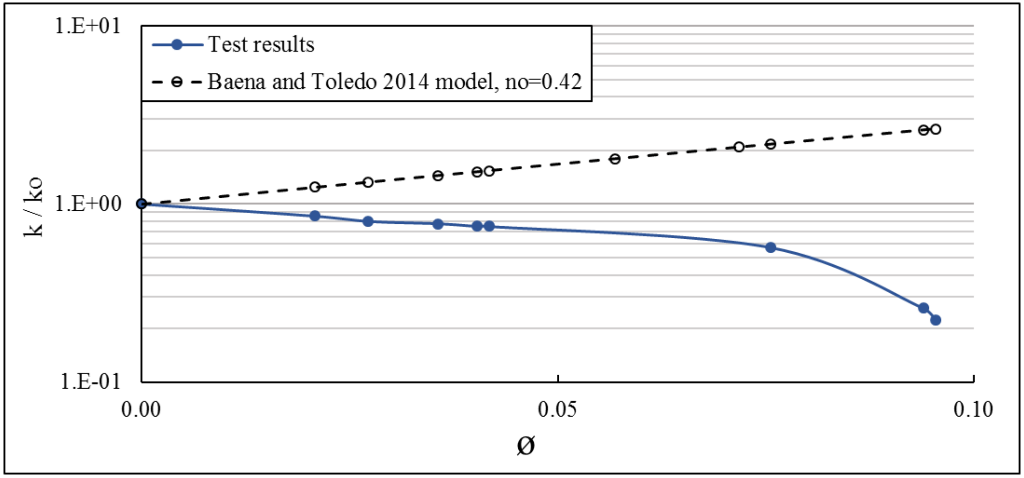
For the test results, the measured k/the initial k was plotted against the values of Ø, whereas the same values of Ø were plugged into the model with an initial porosity of $n_o = 0.39$ for high gypsum soil, and $n_o = 0.42$ for the medium gypsum soil to predict k_{td} / k_o.

The results and the model are both shown in Figure 6.6. It shows that the tests results and the Baena and Toledo (2014) model do not match. The measured permeability for both soils decreased with an increase in Ø, whereas the predicted permeability using the model increased with an increase in Ø.

In their study, Baena and Toledo (2014) stated that when the dissolution rate is very high, a “post-dissolution compaction” will occur in the insoluble solid material, and the model will give an “overestimated k_{td}/k_o ratio”.



High gypsum soil



Medium gypsum soil

Figure 6.6 Baena and Toledo, 2014 model for high and medium gypsum soils.

6.2.1.1.4 New modification on Baena and Toledo, 2014 model

As shown in the previous section, the model did not represent the actual test results, and therefore, to consider the post-dissolution compaction, which will occur in soil with high soluble solids dissolution, a new approach was chosen to modify the Baena and Toledo (2014) model. The new approach was to assume that the volume of the dissolved particles would reduce the porosity (after dissolving), which is the opposite of the Baena and Toledo (2014) assumption.

This assumption was chosen because the results of our study showed that with gypsum dissolution, a reduction in the coefficient of permeability, k, occurred.

$$n = n_o - \emptyset$$

$$k_{td} = k_o \frac{(1-n_o)^2 (n_o-\emptyset)^3}{n_o^3 (1-n_o+\emptyset)^2} \qquad \text{(Eq. 6.5)}$$

To test the modified model with the new assumption, the previous ranges of Ø and n_o, which were used to test the original model, were selected to plot the relationship between k_{td}/k_o and Ø for different values of n_o, which are shown in Figure 6.7.

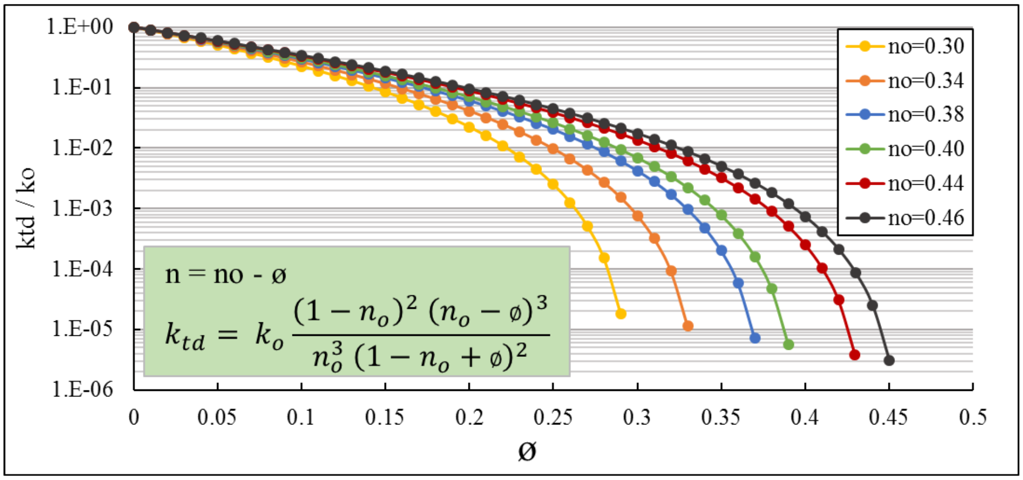


Figure 6.7 The modified Baena and Toledo, 2014 model for a specific range of n_o and Ø.

Applying the Ø and n_o values showed that k_{td}/k_o decreased with an increase in Ø for all the selected n_o values. With the new assumption, the model is more sensitive to Ø changes, which can be seen by comparing the two figures before and after the application of the new assumption. Before the change, the behavior of all the n_o curves was almost the same for Ø values until the value of Ø was approximately 0.4, whereas in the case of the new assumption, the behavior starts to change when the value of Ø is approximately 0.1.

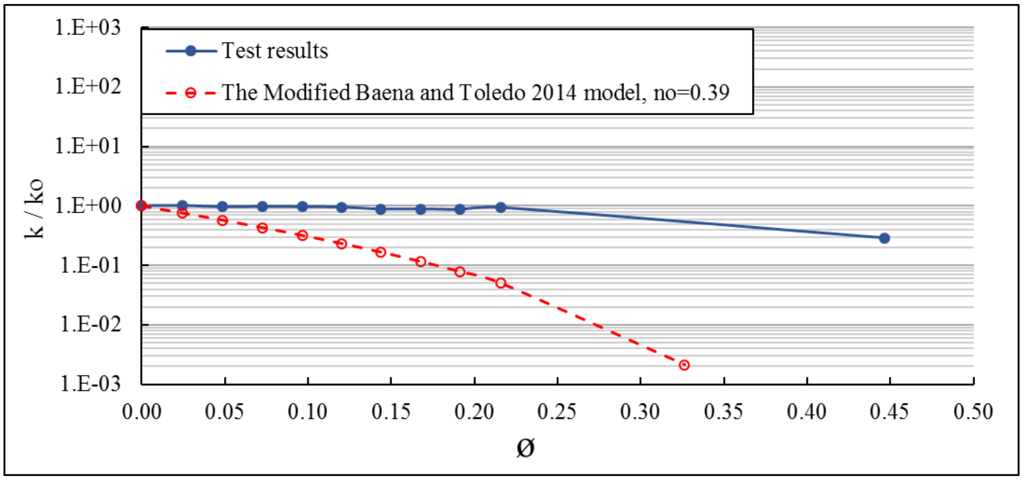
From the relationship of n_o and k_{td}/k_o, it can be inferred that the range of Ø for granular soil is 0 – 0.45. Within this range, k will decrease with an increase of Ø. Moreover, for each n_o curve, the range of Ø was different from other n_o curves. For example, for $n_o = 0.3$, the range of Ø was from 0 – 0.29 because at Ø = 0.3, the value of k_{td}/k_o was equal to zero, meaning that the value of k = 0, which is not feasible.

This model has a limit, which occurred when Ø = n_o because at this value, k = 0 and beyond this value, the values of k_{td}/k_o would be negative. Knowing that V_t is constant, when Ø = n_o, V_s (volume of the dissolved solids) = V_v (the volume of voids in the sample), which means that the volume of the solids that have been dissolved is equal to the volume of voids in the sample.

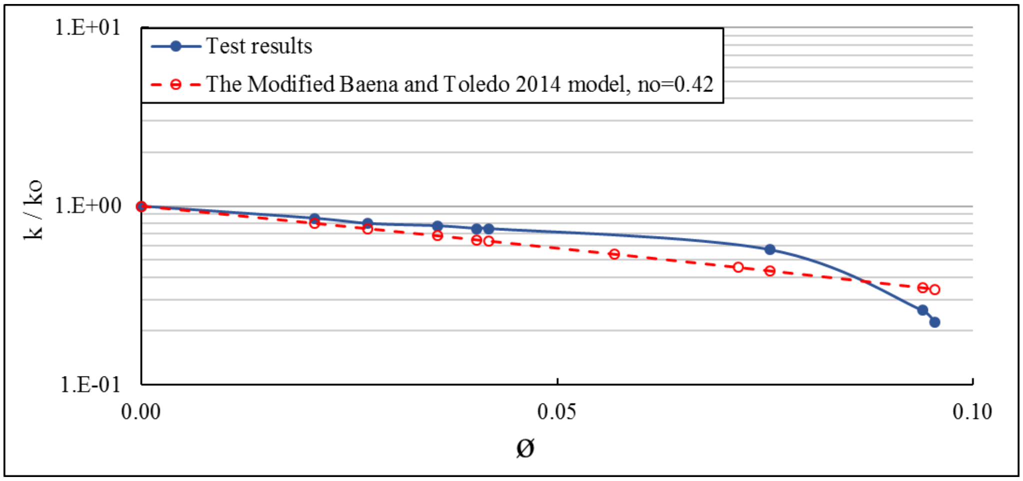
Theoretically, based on the new assumption of n = n_o – Ø, all the voids are filled with solids due to post-compaction, which occurred due to dissolution, n reached a value of zero, and no more voids were in the samples to allow water to flow. This instance would result with a coefficient of permeability that equals zero, which is definitely not feasible.

6.2.1.1.5 Using the modified Baena and Toledo, 2014 model for untreated soils results

The same values of Ø were plugged into the modified model to see if the model provided a close fit to the test results, which is shown in Figure 6.8 for both high and medium gypsum soils. In general, the modified model has a reduction trend, which can be seen from the above figure, and it has a relatively good fit for a small range of Ø (as shown for medium gypsum soil). However, it shows lower values when comparing both test results.



High gypsum soil



Medium gypsum soil

Figure 6.8 Using the modified Baena and Toledo, 2014 model for high and medium gypsum soils.

In the high gypsum soil, the modified model provided a negative value for Ø values of 0.43 and 0.45, which cannot be shown on logarithmic scale. In the case of medium gypsum soil, the gap between the actual results and the modified model was less than that of high gypsum soil. Nevertheless, the modified model did not exactly fit the actual test results. The assumption for this modified model was that all the volume of the dissolved solids (which, in this case, is gypsum) would be filled with insoluble solids.

From Figure 6.8, it can be concluded that this assumption was not 100% accurate because if all the volume of the dissolved solids (Ø) was filled completely with insoluble solids, the modified model would be fit exactly with the actual test results for both soils.

6.2.1.1.6 Account for partial Ø in the modified Baena and Toledo, 2014 model

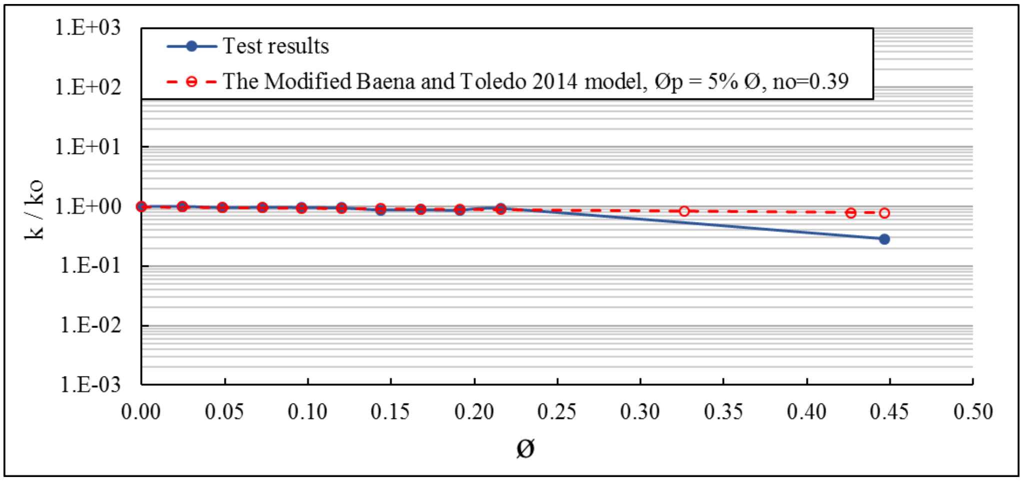
The previous findings indicated that the volume of the dissolved solids was partially filled with insoluble solids, i.e., we cannot subtract the entire Ø from the initial porosity (n_o). Therefore, another modification was done on the model by assuming that a partial value of Ø ($Ø_p$) would be subtracted from the initial porosity (n_o). Different values of $Ø_p$ were plugged into the modified model to see what the value would be, which gives the closest fit for the actual test results for both soils and was plotted in Figure 6.9.

$$\emptyset_p = \%\,\emptyset$$

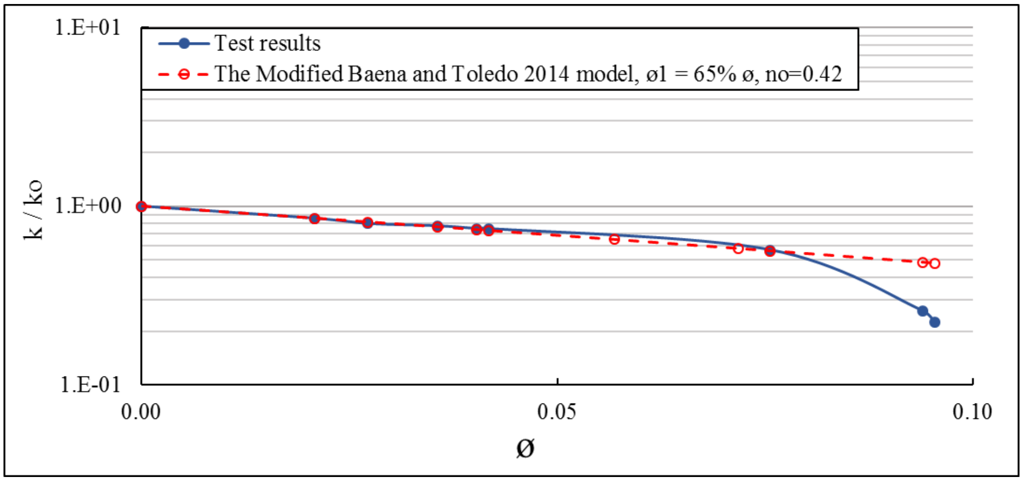
$$n = n_o - \emptyset_p$$

$$k_{td} = k_o \frac{(1-n_o)^2 \left(n_o-\emptyset_p\right)^3}{n_o^3 \left(1-n_o+\emptyset_p\right)^2} \qquad \text{(Eq. 6.6)}$$

The test data using the new modification fits the model well in both soils, as can be seen from Figure 6.9. In the case of high gypsum soils, the model shows that only 5% of the volume of the dissolved and leached solids (gypsum) was filled with insoluble solids and participated in the reduction of the k value. however, the self-collapsing of the soil sample, which occurred due to the very high rate of dissolution, was the major contributing factor to the reduction of the coefficient of permeability. For medium gypsum soil, the behavior was slightly different.



High gypsum soil



Medium gypsum soil

Figure 6.9 Using the modified Baena and Toledo, 2014 model with $Ø_p$ for high and medium gypsum soils.

In this case, the model revealed that 65% of the volume of the dissolved solids was filled with the insoluble solids, in addition to the self-collapsing of the soil sample. Both of these factors resulted in the reduction of the coefficient of permeability.

6.2.2 Treated soils test

6.2.2.1 First trial

In this trial, the same constant head cell was used during the test. High and medium gypsum samples were prepared by mixing the soil with 6% asphalt emulsion, curing, and then compacting in the permeameters. Figure 6.10 shows the relationship of the k value and the leachate volume.

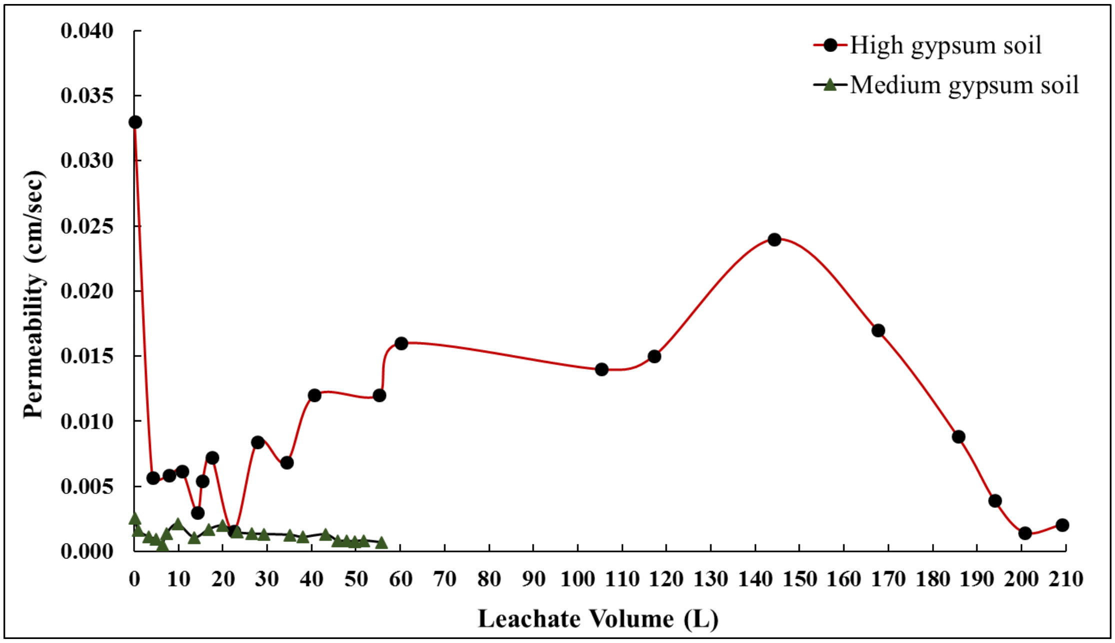


Figure 6.10 Coefficient of permeability for high and medium gypsum soils treated with 6% asphalt emulsion.

The k value in high gypsum soil fluctuated with the leachate, but toward the end, it has a decreasing trend. Whereas, the k value was relatively constant in medium gypsum soil, although much lower than the high gypsum soil, with Lower amounts of water flowed through sample within the same time period. EC measurements are shown in Figure 6.11.

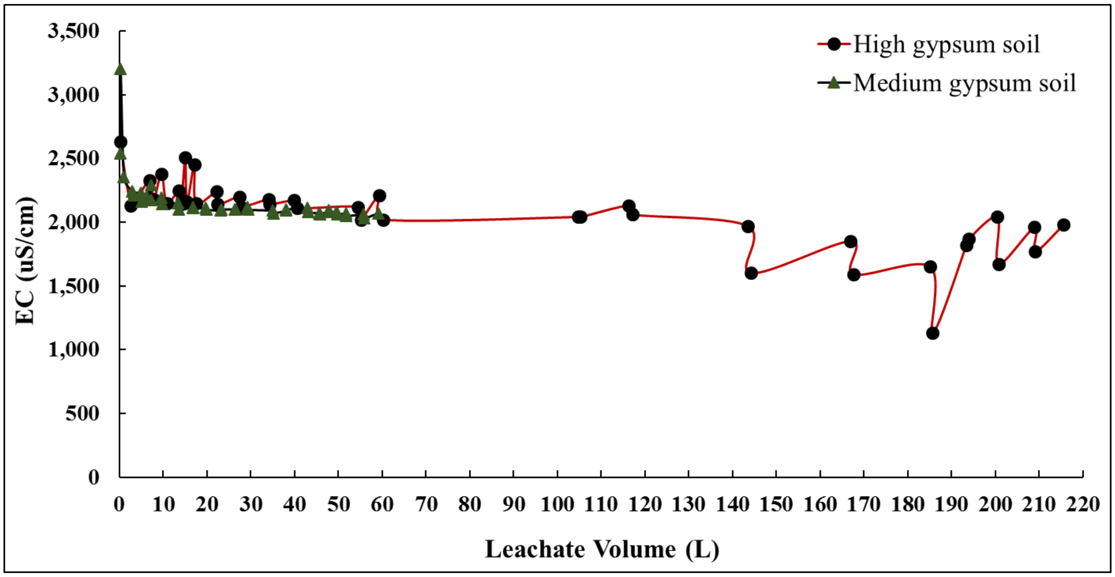


Figure 6.11 EC for high and medium gypsum soils treated with 6% asphalt emulsion.

Less EC fluctuation was observed in medium gypsum soil compering to the high one. Medium gypsum soil had relatively EC of approximately 2000 uS/cm. While the EC values for though high gypsum soil experienced greater variability, but it was also approximately 2000 uS/cm. No decrease in the EC with water flowing was observed. With higher amounts of water flowing through the high gypsum soil, these values were used to estimate the gypsum dissolution as shown in Figures 6.12 and 6.13.

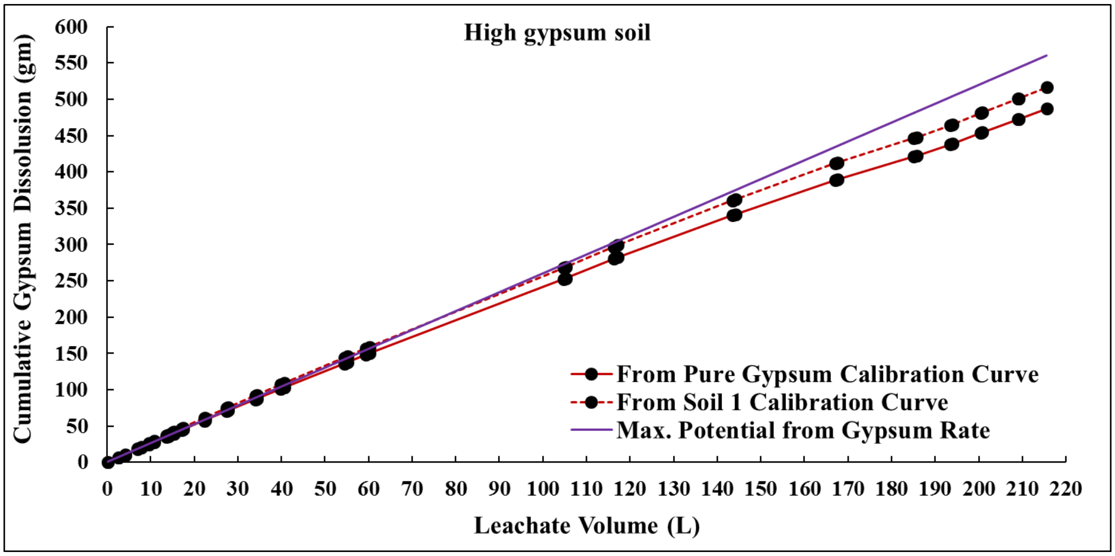


Figure 6.12 Gypsum dissolution for high gypsum soil treated with 6% asphalt emulsion.

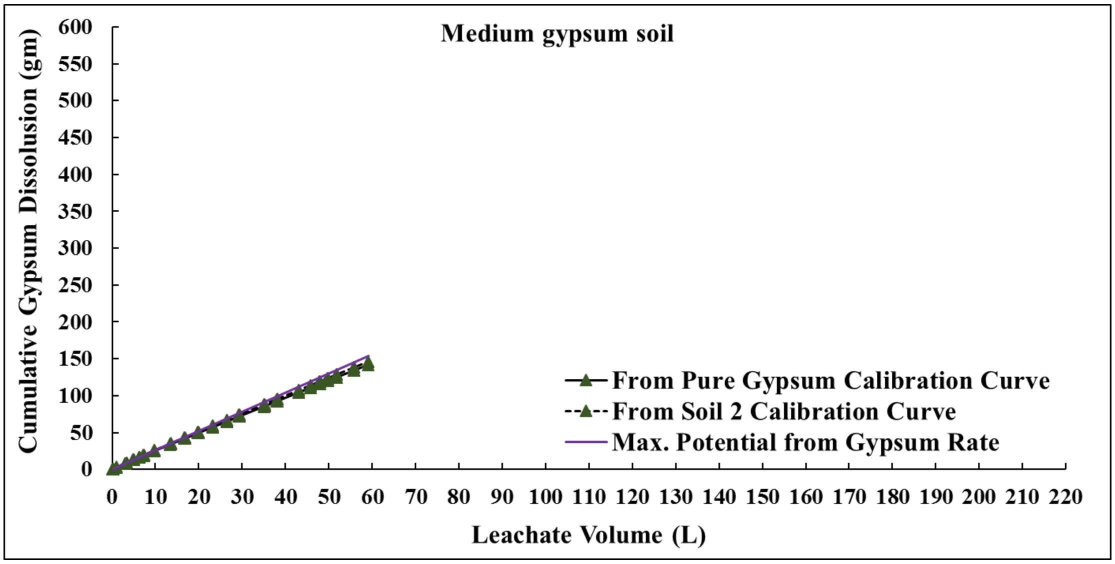


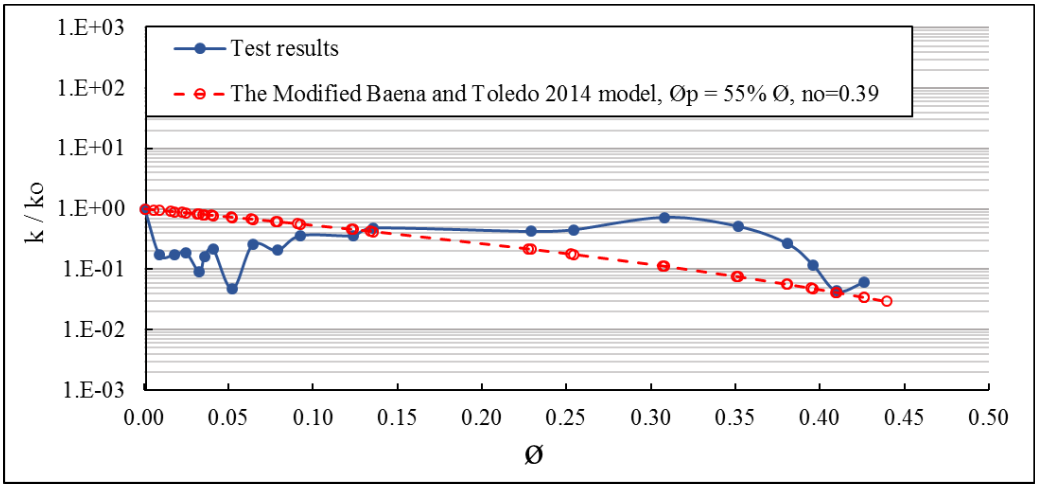
Figure 6.13 Gypsum dissolution for medium gypsum soil treated with 6% asphalt emulsion.

In high gypsum soil, Figure 6.12 shows that treatment by mixing, curing, and then compacting the sample in the cell provided a slight reduction in gypsum dissolution towards the end of the test by comparing the two dissolution estimates with the maximum potential rate.

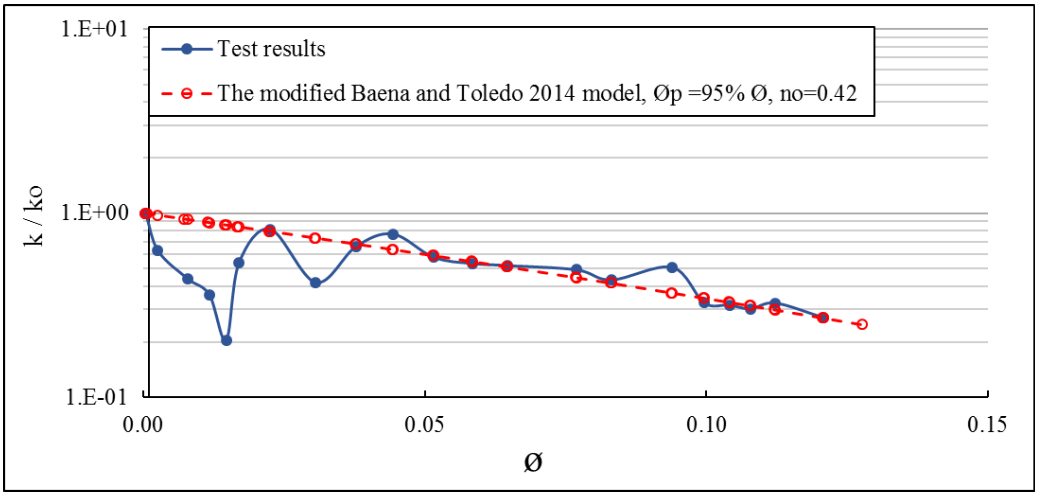
For medium gypsum soil (Figure 6.13), the results showed that the dissolution was lower than high gypsum soil, but the rate of the dissolution was the same as the maximum gypsum rate until the end of the test.

6.2.2.1.1 Using the modified Baena and Toledo, 2014 model for first trial samples

To investigate the effectiveness of the first trial treatment on the coefficient of permeability for both treated soils, the modified Baena and Toledo (2014) model ($Ø_p$ modification) was plotted against Ø values for both soils (high and medium gypsum soils). The test results represent the actual k/k_o values, whereas the modified model was used to calculate the theoretical k/k_o values, as shown in Figure 6.14.



High gypsum soil



Medium gypsum soil

Figure 6.14 Using the modified Baena and Toledo, 2014 model with Ø$_{p}$ for trial one treated high and medium gypsum soils.

For the first trial treatment, the modified model also was almost matched the actual test values but with different Ø$_{p}$ values in high gypsum soil and medium gypsum soils. The value of Ø$_{p}$ that was plugged into the modified model for high gypsum soil was approximately 55% of Ø, which provided a curve that represented the average test values, as shown in Figure 6.14. This results suggests that approximately 55% of the dissolved solids were filled with insoluble solids, which resulted in reductions in the porosity and the coefficient of permeability.

On the other hand, a value of $Ø_p$ of approximately 95% of the Ø provided a model fit that is almost exactly the same as the actual test results, which suggests that approximately 95% of the volume of dissolved solids was filled with insoluble solids and reduced the intergranular voids and the coefficient of permeability.

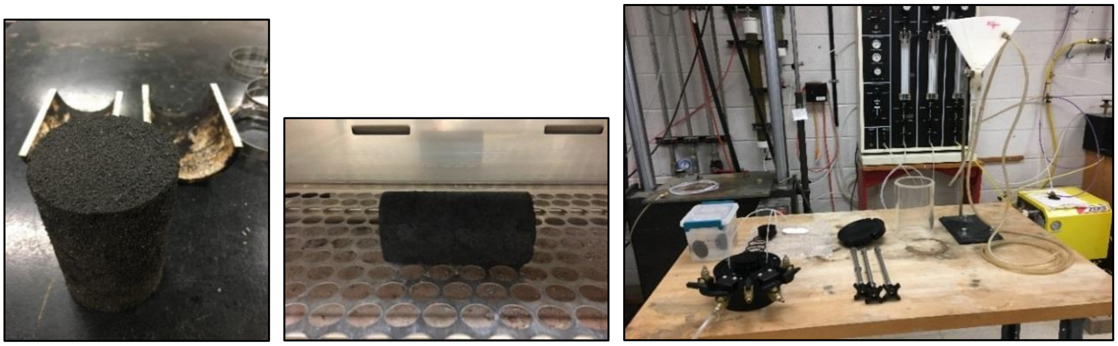
Nevertheless, the high rate of dissolution and the post-dissolution collapsing was the most responsible factor in void reduction for high gypsum soil, while in the medium gypsum soil, the filling of the volume with insoluble solids was the major factor, which reduced the coefficient of permeability.

Overall, these findings indicated that using the Trial 1 approach to treat both soils was not a good option to prevent dissolution. Although there was a reduction in permeability, this reduction was related to the post-dissolution compaction and collapsing, which was also seen in the untreated soil tests.

6.2.2.2 Second trial

Based on the first trial results, and since the approach of mixing, curing, and compacting did not mitigate gypsum from dissolving, a flexible wall permeameter was used by combining the flexible wall ASTM D5084-16a standard (for confining the sample only) and the constant head ASTM D 2434 – 68 standard for constant head permeability testing.

In this case, the samples were mixed with 18% asphalt emulsion, compacted, cured, and then placed in a membrane inside the cell, as can be seen in Figure 6.15. The same measurements were also taken, Figure 6.16 shows the k value versus the leachate volume.



Molding Curing Permeability cell

Sample setup Confining pressure

Figure 6.15 Soil sample molding, curing, and setup.

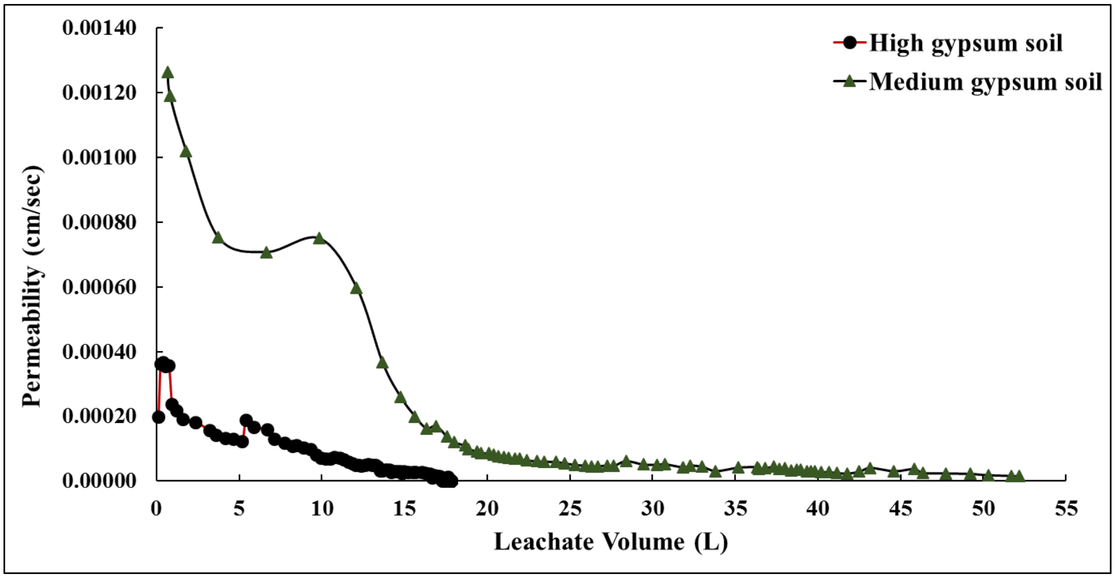


Figure 6.16 Coefficient of permeability for high and medium gypsum soil treated with 18% asphalt emulsion (flexible wall method).

In this type of treatment, medium gypsum soil started with a k value that was higher than the high gypsum soil. The k value decreased in both soils with an increase in water flow, but in this case, high gypsum soil prevented more water from flowing through the system, compared to medium gypsum soil. Towards the end of the test, no more water leached out from the high gypsum soil sample. These results were linked to the fact that there were physical changes that occurred in the soil structure between the gypsum particles and the asphalt binders. The more available gypsum in the soil, resulted in more these physical attraction, as shown in Fan et al. (2019).

They found that mixing different amounts and types of calcium sulfate whiskers with different types of asphalt binders improved the workability and the strength of the binders. These improvements increased with an increase in the calcium sulfate whiskers. The EC measurements are shown in Figure 6.17. It can be seen that high gypsum soil started with high EC and then reduced to a constant value. However, towards the end, the EC fluctuated with a very limited amount of water leaching of the sample.

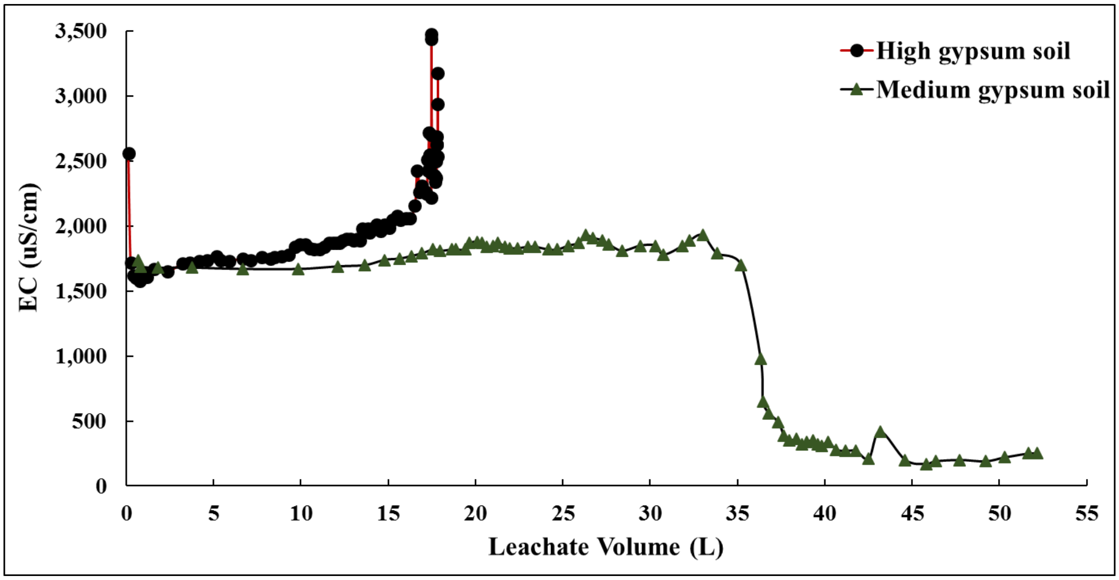


Figure 6.17 EC for high and medium gypsum soil treated with 18% asphalt emulsion (flexible wall method).

The medium gypsum soil achieved a constant value until approximately 35L of leachate. Then, a sudden drop in the EC occurred. Afterwards, the EC showed a constant trend. Gypsum dissolution was estimated from the EC and is shown in Figures 6.18 and 6.19. For high gypsum soil, even though gypsum dissolves with water flow, the dissolution was less than the maximum potential that was predicted from the general gypsum dissolution rate.

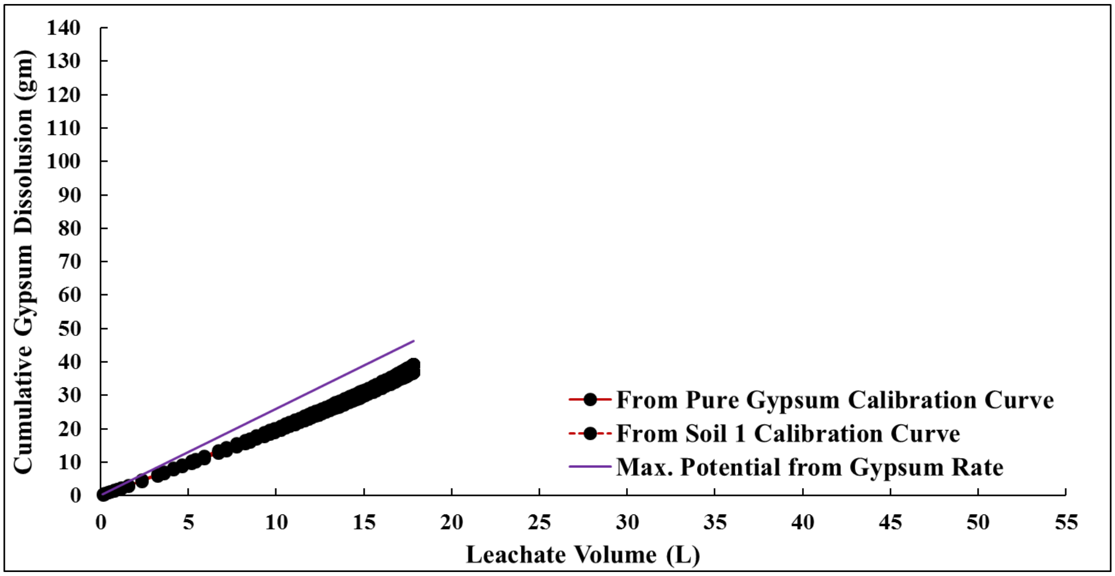


Figure 6.18 Gypsum dissolution for high gypsum soil treated with 18% asphalt emulsion (flexible wall method).

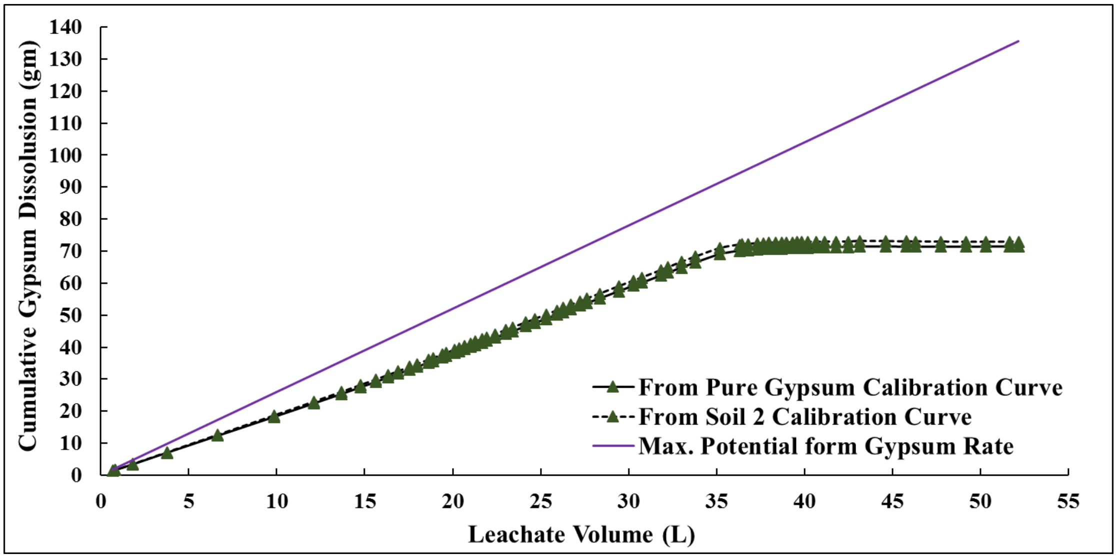


Figure 6.19 Gypsum dissolution for medium gypsum soil treated with 18% asphalt emulsion (flexible wall method).

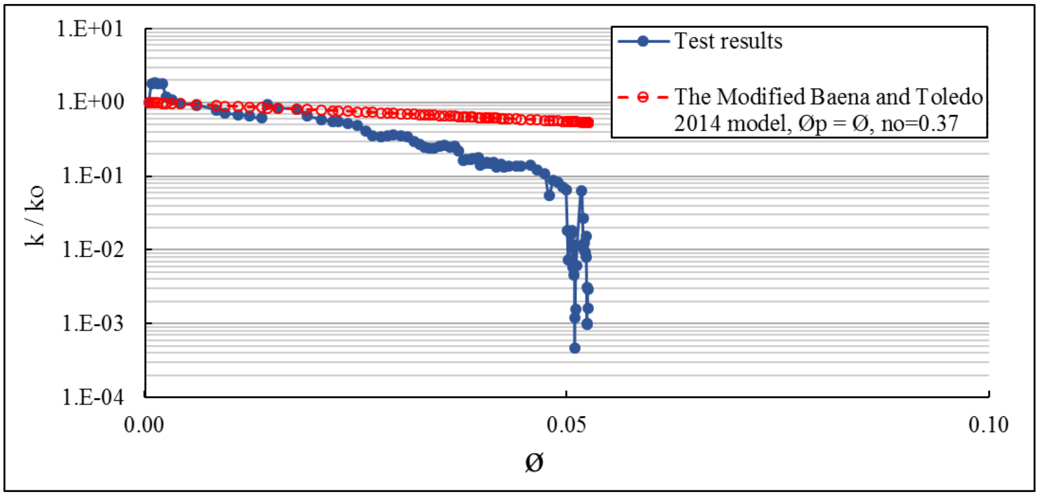
In medium gypsum soil, the asphalt emulsion prevented the gypsum from dissolving, according to the gypsum rate, but this improvement did not start from the beginning, as shown by the cumulative gypsum dissolution. After 36.5L of leachate, some gypsum dissolved, but it was less than 0.5 g in each individual measurement.

Many different types of treatments for various soil types with different amounts of gypsum showed reduction in soil permeability reduced. Aziz and Ma (2011) treated two types of soils with fuel oil: clayey soil with gypsum content of approximately 26% and sandy soil with gypsum content of approximately 51%. In both soils, they found a reduction in the permeability and water leaching, which was related to the rearrangement of the soil particles and resulted in closuring of more voids due to the lubrication effect of the fuel oil.

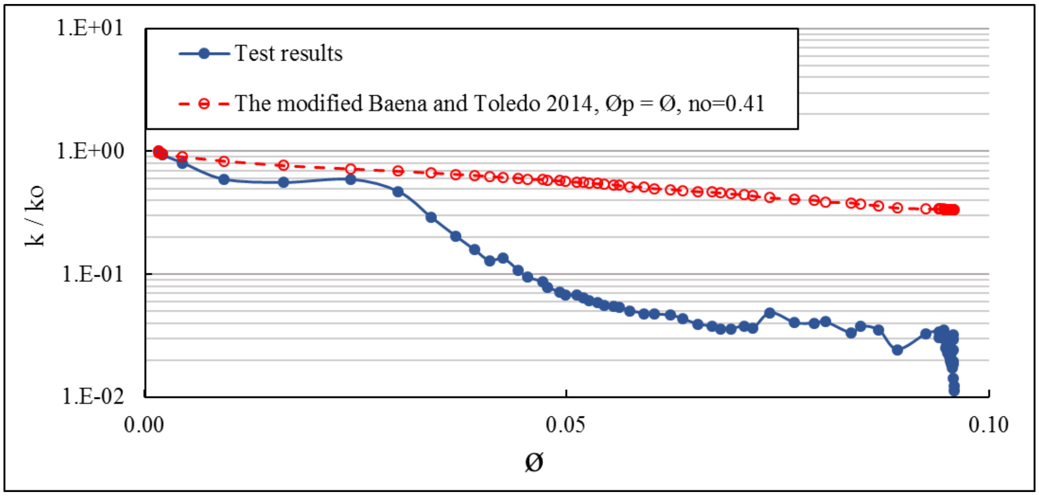
Alsafi et al. (2017) found that treating silty sand, which has a gypsum content of approximately 13%, with activated fly ash will reduce the soil permeability. More activated fly ash resulted in greater reduction in the coefficient of permeability. This result was related to the modifications in the microstructure system of the soil and the reduction in the soil’s voids due to the formation of the geopolymers gels and ettringite.

6.2.2.2.1 Using the modified Baena and Toledo, 2014 model for second trial samples

Figure 6.20 shows the use of the modified Baena and Toledo (2014) model for the second trial treatment for both soils. In this case, the highest $Ø_p$ value, which was 100% of the actual Ø value, was plugged into the modified model for both soils samples. In this case, the use of the modified model showed that this approach was the most appropriate to treat these soils.



High gypsum soil



Medium gypsum soil

Figure 6.20 Using the modified Baena and Toledo, 2014 model with $Ø_p$ for trial two treated high and medium gypsum soils.

The results of the gypsum dissolution measurements previously revealed that this approach was very good in reducing gypsum dissolution, and these results clearly show that the reduction in the coefficient of permeability was not related to gypsum dissolution or the internal rearrangement of the soil structures. These results show that the adhering and bonding between the calcium sulfate molecules and the asphalt binders was the major factor that controlled the reduction in permeability.

As shown in Figure 6.20, even when the entire Ø values were plugged in for both soils, the modified model provided values that were higher than the actual k/k_o curves, which means that the use of the asphalt emulsion improved the soil structure much more than the previous trial, by lowering the volume of voids and the porosity to a magnitude that changed the soil structure from sand to silt or even to clay, based on the coefficient of the permeability.

6.3 Summary and conclusions

This work was performed in two sections. The first section covers the constant head permeability test for two untreated samples: high and medium soils with gypsum. The test included leachate collection and gypsum dissolution measurements.

The second section covered the effect of moving water on the coefficient of permeability and gypsum dissolution for high and medium gypsum soils treated with 6% and 18% asphalt emulsion and cured at 35°C until they reached a constant weight.

The 6% treatment was performed by using the constant head procedure, whereas the 18% treatment was performed using a modified procedure by placing the sample in flexible wall permeameter with a confining pressure of 5 psi. However, the permeability test was performed by using the constant head method procedure with a head of 17.8 cm. The leachates were also collected for gypsum dissolution measurements.

1- Water flow has a significant impact on gypsum dissolution, but this dissolution reduces the coefficient of permeability with time, since gypsum dissolution changes the soil structure, and the movement of the large gypsum particles will close the micro tubes inside the soil.

2- The treatment approach affects the water flow and gypsum dissolution, which was clear when the approach of mixing, curing and then compacting was used. This approach did not change the amount of gypsum dissolution, compared to the test on the untreated samples.

3- Using the approach of mixing, compacting, and curing had a clear effect on the reduction of gypsum dissolution, each soil exhibited different behavior.

  - In high gypsum soil, this approach reduced gypsum dissolution, not by preventing it from dissolving, but by reducing the k value such that there was no flow toward the end of the test.
  - In medium gypsum soil, this approach prevented gypsum dissolution by reducing the amount of gypsum that leached from the sample. However, this prevention did not start from the beginning because the value of EC started high and remained constant until approximately 35L of water flowed through the sample. Afterwards, a sudden drop occurred in the EC.

4- Using the modified Baena and Toledo (2014) model helped to predict what occurred in the microstructure of the soil when the asphalt emulsion was used in both trials and how the second trial approach improved the reductions in gypsum dissolution and the coefficient of permeability.

Chapter 7

A Comparison Between the Unconfined Compressive Strength (UCS) for Gypseous and Gypsiferous Sandy Soils Treated with Activated Fly ash, Asphalt Emulsion, and Portland Cement

7.1 Materials and methods

7.1.1 Soils

High and medium gypsum poorly graded sand soils (S1 and S2), as described in previous chapters were used in this study.

7.1.2 Activated fly ash

Class F Fly ash activated with 12 M potassium hydroxide (KOH) solution, as described in Chapter 4, was used for fly ash treatment.

7.1.3 Asphalt emulsion

Slow setting NTSS-1HM, produced by BLACLIDGE EMULSION, Inc. (Greer, South Carolina, USA), as described in Chapter 5, was used for asphalt emulsion treatment.

7.1.4 Portland cement

Type I/II Portland cement was used for cement treatment.

7.1.5 Specimen preparation and curing

The preparations and curing for specific specimens used in the wetting-drying tests, according to the ASTM D559/D559M – 15 standard were described in Chapters 4 and 5 for specimens for activated fly ash, asphalt emulsion, and Portland cement treatments.

7.1.6 Unconfined compressive strength (UCS) test

The procedure outlined in the ASTM D2166/D2166M – 16 standard was used to measure the UCS for N1 specimens that survived all the wetting-drying cycles, according to ASTM D559/D559M – 15. A Jeo Jac automated device was used, with a strain rate of 1%/min. (1mm/min.).

7.1.7 Young's Modulus (E)

The secant modulus of elasticity was used to estimate the modulus of elasticity from the stress-strain relationship in each case. The measurements were done in a range of 30-70% of the maximum strength to ensure that the readings were taken within the elastic zone.

7.2 Results and discussion

7.2.1 Unconfined compressive strength (UCS) and Young's Modulus (E) for specimens treated with activated fly ash

As shown in Chapter 4, few specimens from both soils survived the twelve wetting-drying (W-D) cycles.

Table 7.1 shows the UCS and E values for these specimens. Figure 7.1 shows the only specimen for high gypsum soil that survived the W-D cycles, which was treated with 30% activated fly ash and cured for 28 days.

Table 7.1 UCS and E for W-D survived specimens (activated fly ash).

Specimen No.	7 days curing		28 days curing	
	UCS (kPa)	E (MPa)	UCS (kPa)	E (MPa)
S1,F30%,N1	---	--	973.36	104.94
S2,F20%,N1	----	------	170.83	48.37
S2,F30%,N1	635.29	34.16	371.48	27.87

F%: Activated fly ash percentage.

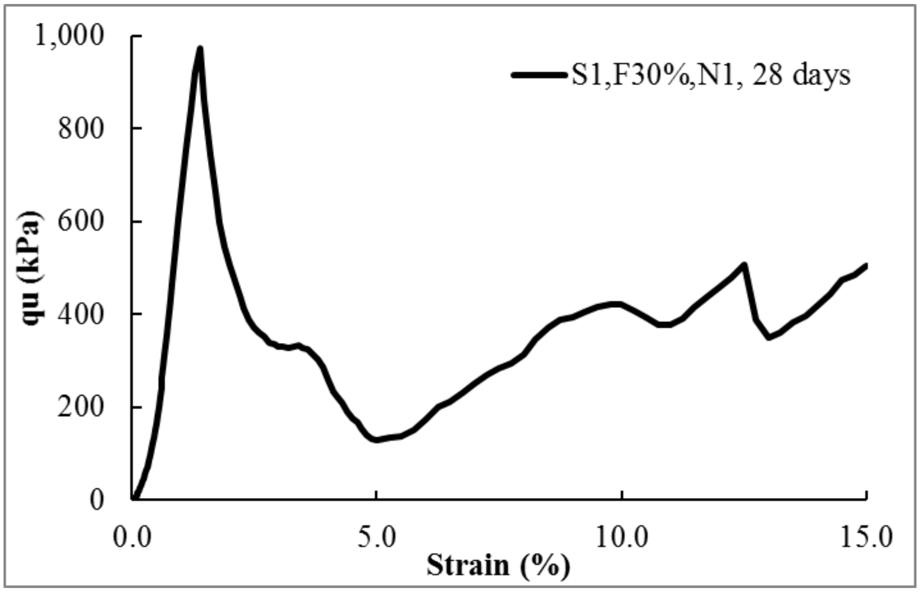


Figure 7.1 UCS for high gypsum soil specimen treated with 30% activated fly ash and cured for 28 days.

Medium gypsum soil has three specimens that survived the W-D cycles. For an activated fly ash percentage of 20%, the specimen that was cured for 28 days, shown in Figure 7.2, survived the W-D cycles, and for the 30% activated fly ash treatment, both specimens that were cured for 7 and 28 days survived the W-D cycles, as shown in Figure 7.3.

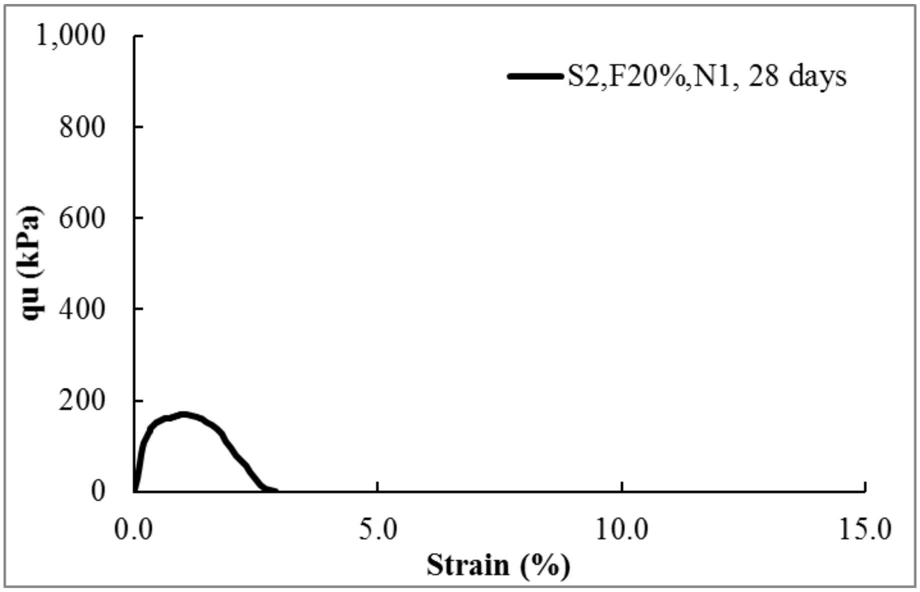


Figure 7.2 UCS for medium gypsum soil specimen treated with 20% activated fly ash and cured for 28 days.

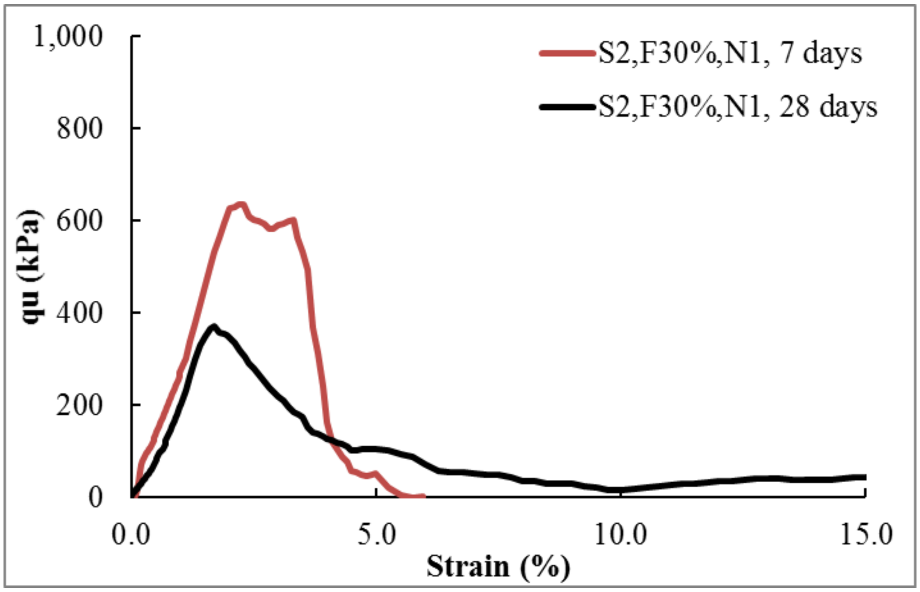


Figure 7.3 UCS for medium gypsum soil specimens treated with 30% activated fly ash and cured for 7 and 28 days.

As shown in the figures above, higher gypsum soil provided the highest compressive strength and the highest Young’s Modules. The failure happened suddenly with a very steep curve, which suggests that the treatment of highly gypsum soil with activate fly ash behaved as a stiff material (brittle failure mode).

While the relationship between the activated fly ash and the compressive strength for medium gypsum soil showed that the strength generally increases with increased activated fly ash increase, the results of the specimens that were treated with 30% activated fly ash showed that a decrease in strength with an increase in curing period, as shown in Figure 7.3. In this study, the formation of potassium aluminosilicate hydrate K-A-S-H gel by activating Class F fly ash with potassium hydroxide (KOH) enhanced the strength of both soils. The formation of the geopolymer gel (i.e., aluminosilicate hydrated A-S-H or sodium aluminosilicate hydrate N-A-S-H) inside the soil voids resulted in more compacted microstructures, which improved the overall strength of the soil (Van Deventer et al., 2015; Alsafi et al., 2017).

Higher gypsum content in the soil provides the highest compressive strength, as can be observed by comparing high gypsum soil with the medium gypsum soil for specimens that were treated with 30% activated fly ash and cured for 28 days. This result is related to the formation of ettringite ($(CaO)_3(Al_2O_3)(CaSO_4)_3 \cdot 32H_2O$). Even at early stages of curing, gypsum, which is $CaSO_4.2H_2O$, is the source of calcium sulfate ($CaSO_4$), and the Class F fly ash used in the study is the source of calcium aluminate because it has approximately 21.2% aluminum oxide (Al_2O_3), and 5.3% of calcium oxide (CaO).

From mixing a range of gypsum dosage (1-6%) with lime-treated sand, Jha and Sivapullaiah, (2017) found that the sulfate (from gypsum) consumption process during ettringite formation also leads to the production of silica (from the sand and the fly ash).

More silica results in the formation of additional calcium-silicate-hydrate (C-S-H) gel and calcium-aluminate-silicate-hydrate (C-A-S-H) gel due the use of the alkaline activated fly ash. They also proved that ettringite formation results in a more compacted and reinforced structure in the sand that was treated with activated fly ash, which provides more soil strength.

However, they found that soil strength was controlled by the amount of gypsum in the soil and the curing period. The formation of additional ettringite that exceeds the amount of voids in the soil will lead to an expansion of the soil structure and a reduction in its strength.

In this study, a reduction in the compressive strength occurred in medium gypsum soil when additional curing was time used, which was related to more ettringite had been formed, such that it exceeded the volume of the voids/pores in the soil (Figure 7.3).

7.2.2 Unconfined compressive strength (UCS) and Young's Modulus (E) for specimens treated with asphalt emulsion

Three different percentages (6%, 12%, 8%) of anionic slow setting asphalt emulsion were used to treat both sandy gypsum soils, and the specimens were subjected to W-D cycles, as discussed in Chapter 5. The unconfined compressive strength (UCS) was measured, and the Young's Modulus (E) was calculated for each test. Table 7.2 shows the UCS and E results for all specimens that survived all twelve cycles. Figures 7.4 and 7.5 show the results of the unconfined compressive strength (UCS) for both high and medium gypsum soils that were treated with asphalt emulsion.

Table 7.2 UCS and E for W-D survived specimens (asphalt emulsion).

Specimen No.	UCS (kPa)	E (MPa)
S1,6%,N1	200.47	18.21
S1,12%,N1	701.33	35.04
S1,18%,N1	1353.34	47.14
[1]S2,6%,N1	-------	-------
S2,12%,N1	543.51	40.06
S2,18%,N1	970.80	50.81

%: Asphalt emulsion percentage.

[1] This specimen did not survive (failed in cycle nine).

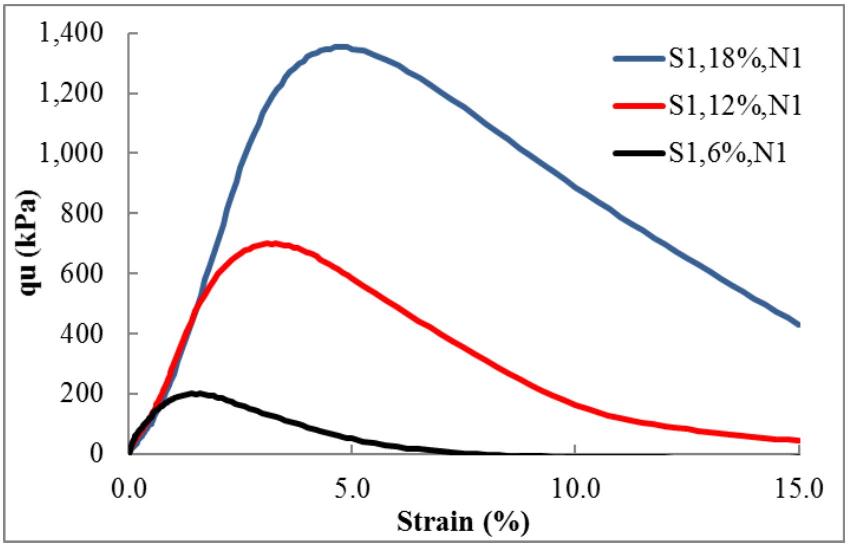


Figure 7.4 UCS for high gypsum soil's specimens treated with asphalt emulsion.

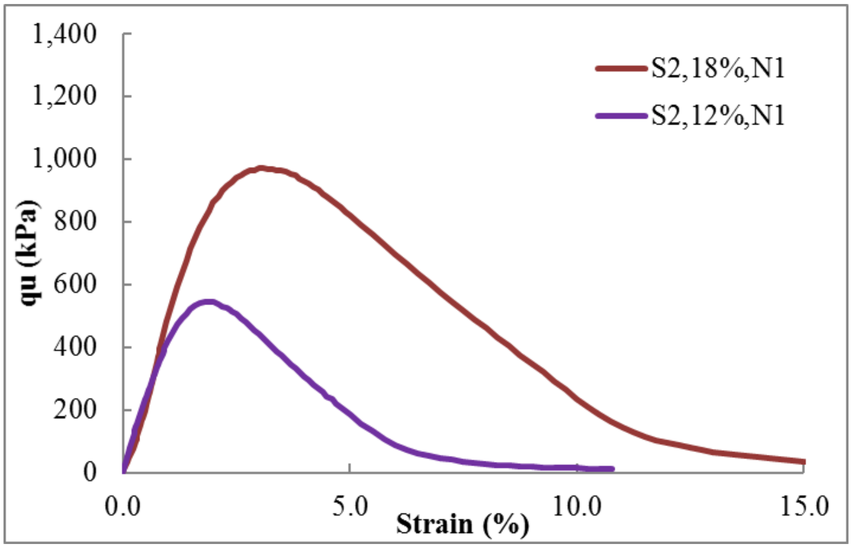
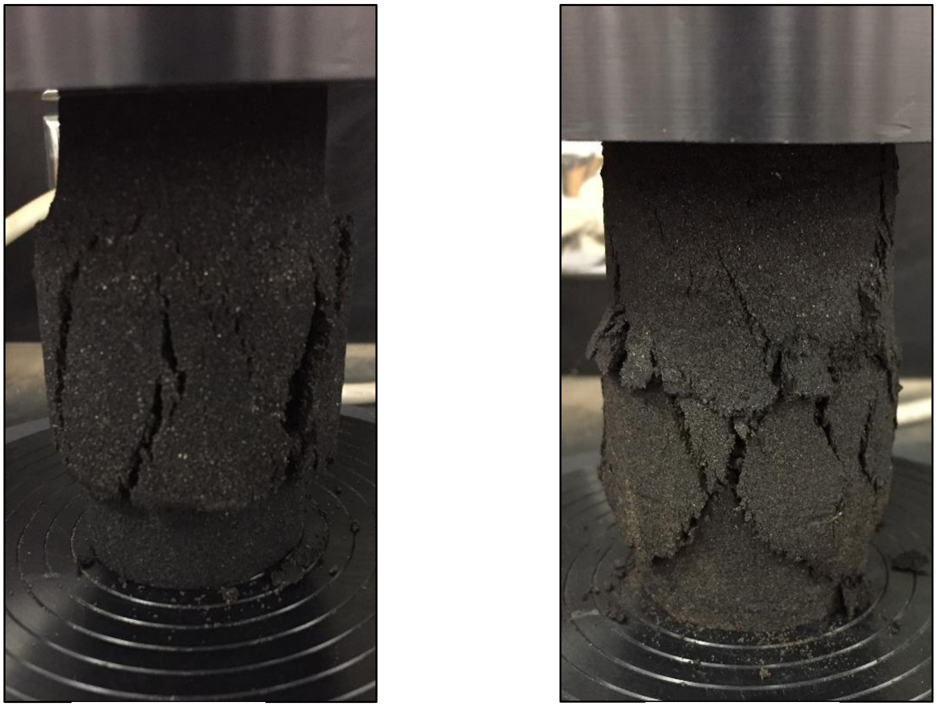


Figure 7.5 UCS for medium gypsum soil's specimens treated with asphalt emulsion.

From the figures above, it can be seen that the correlation between the unconfined compressive strength (UCS) and the asphalt emulsion percentage was positive. Moreover, by comparing both soil results, it is clear that with the increase in gypsum content, the UCS also increases for each asphalt emulsion percentage.

Also, by observing the failure type for each specimen, it can be determined that the failure mode is semi-plastic, as shown in Figure 7.6, which shows failure mode examples for two specimens.



S1,12%,N1

S2,18%,N1

Figure 7.6 UCS failure mode for high and medium gypsum soils' specimens treated with asphalt emulsion.

Taha et al. (2008) and Ahmed (2014) used cut-back RC-70 and asphalt emulsion, respectively, to treat different types of soils. They found an optimum asphalt emulsion content, at which point the soil strength began to decrease.

In this study, the strength increased with an increase in gypsum content (by comparing both high gypsum soil with medium gypsum soil) for each asphalt emulsion percentage. No previous study that showed these close observation was found until development of this work.

However, a study conducted by Fan et al. (2019) investigated the effects of mixing three different types of calcium sulfate whiskers with two different types of asphalt binders on the performance of asphalt binder. They showed that modifications were made on the asphalt binders when mixed with calcium sulfate whiskers.

In the dynamic shear rheometer (DSR), which is a test that is used to find the shear modulus (G) and the phase angle (δ) to calculate the rutting factor (G/Sin(δ) for both types of asphalt binders treated with three different types of calcium sulfate whiskers, they proved that the rutting factor, which is a property that shows the rutting resistance performance (i.e., higher rutting factors suggest higher rutting resistance), increased with an increase in calcium sulfate content.

Moreover, the bending test rheometer (BBR) was used to evaluate the crack resistance performance (the creep stiffness S) of the modified asphalt by measuring the bending moment under low temperature. Their results showed that higher calcium sulfate content yielded highest creep stiffness S.

Although higher S values result in lower temperature crack resistance, the stiffness of the binders continued to increase with an increase in calcium sulfate content. To investigate the microstructure changes in the asphalt binders, they used the following tests:

The use of X-Ray Diffraction (XRD) proved that there was no formation of new crystalline phases due to the mixing of asphalt and the calcium sulfate whiskers and that all the effects of the modifications effects were related to the physical properties that were represented by the “cross-link” of the whiskers into the binders to form a new structure to resist the external forces.

This result has also been supported by the findings of another test called the Fourier transform infrared (FTIR). This test showed that all the modifications were related to physical binding and not any chemical reactions. The last test was the Scanning Electron Microscopy (SEM) images, which showed that the calcium sulfate whiskers created a “network-reinforced structure” with the asphalt binder by distributing it in different directions and locations inside the binder network.

They explained that this result was related to the absorption process, which occurred at the rough surfaces of whiskers. These whiskers absorb the light oil of the binder, the steric acid coupling agent, and the silane coupling agent from the binders, which create very high adhesion between the binder and the whisker.

The results of Fan et al. (2019) state that higher calcium sulfate content mixed with the asphalt provided higher strength in the modified asphalt, which was related only to the physical mechanism and not any chemical reactions. These results support our study because we found that higher gypsum content provided higher UCS, which was related to interlocking and adhesion that occurred in the microstructure phase between the asphalt binders and the calcium sulfate molecules.

7.2.3 Unconfined compressive strength (UCS) and Young's Modulus (E) for specimens treated with Portland cement type I/II

As discussed in Chapter 5, N1 and N2 specimens of sandy soils with gypsum treated with 9% Portland cement type I/II and mixed with 9% water content were prepared and used as reference specimens because the wetting-drying ASTM D559 standard was designed for soils treated with cement. These specimens were also subjected to twelve wetting-drying cycles, and all of them survived. N1 specimens for both soils were placed in a Wykeham Farrance (WF) compression test machine to measure the unconfined compressive strength (UCS).

In this case, the Jeo Jac automated device was not used because during the test of the high gypsum soil specimen, it reached its capacity without failure. Table 7.3 shows the UCS results for the cement treated specimens.

Table 7.3 UCS and E for W-D survived specimens (Portland cement).

Specimen No.	**UCS (kPa)**	**E (MPa)**
S1,9%,N1	5052.06	412.14
S2,9%,N1	2243.84	-------

Figure 7.7 shows that the failure mode in both specimens was a brittle failure. The failure planes occurred close to the top and on the outer edges of the specimens. The results of the cement treatment showed that the strength of high gypsum soil was higher than (approximately double) the medium gypsum soil. Moreover, the failure mode showed that on both soils, the core of the specimen was harder than the outside, which can be seen as the failure started at the specimen circumference.

S1,9%,N1

S2,9%,N1

Figure 7.7 UCS failure mode for high and medium gypsum soils' specimens treated with Portland cement.

As discussed earlier, these specimens went through twelve cycles of wetting-drying, and these cycles improved their strength due to the continued process of cement hydration. The increased strength was related to the formation of ettringite and/or thaumasite and these hydrous minerals were primarily related to the availability of water (hydration process).

Moreover, the presence of gypsum (which works as an activator for the pozzolanic reaction in the soil structure) increased the formation of ettringite, along with both the CSH and CASH gels (Jha and Sivapullaiah, 2017). Thus, the more gypsum content in the soil, the highest the strength will be, which was exactly what was found by comparing the results of both soils.

7.3 Summary and conclusions

This chapter discussed a comparison between the unconfined compressive strength (UCS) for high and medium gypsum sandy soils treated with different additives, which include activated fly ash, asphalt emulsion, and Portland cement.

The effects of each individual additive on the UCS for the treated specimen was investigated, and the relationship between gypsum content and UCS also studied. The modulus of elasticity (E) for each case was estimated from the UCS results.

1- By comparing the results, it is obvious that the heights UCS was achieved by treating both soils with Portland cement. However, the use of cement to treat soil with gypsum leads to sulfate attack, particularly during wetting, due to the availability of the sulfate ion from gypsum. This process will degrade the matrix of the stabilized materials and result in large voids and high permeability, which may lead to more gypsum dissolution, higher compressibility, and collapsible potential in the soil (Alsafi et al., 2017).
2- The use of activated fly ash is a good alternative to cement to treat soil with gypsum, and in this study, it had a good stabilization properties with sandy soils, even for very high gypsum soil (93%). Moreover, the strength of the soil increased with an increase in the gypsum content. The problem with this treatment was the continued formation of ettringite with the increase in the curing period. In the field, this process will continue as long as there is available moisture in the soil.

   The formation of additional ettringite that exceeds the volume of voids in the soil structure will result in expansion, soil structure deterioration, and strength reduction. As shown in Chapter 5, very few specimens treated with activated fly ash survived the wetting-drying cycles.
3- Finally, the use of asphalt emulsion showed very good resistance for the wetting-drying cycles. Almost all the specimens survived all the twelve cycles.

For the UCS of the soil, the use of the asphalt emulsion improved the soil strength (i.e., the higher asphalt emulsion produced the highest soil strength). This increase in strength was related to physical binding and interlocking, which resulted in very high adhering forces between the calcium sulfate molecules from gypsum and the asphalt binders, after reaching the coalescence stage. From all of the above, it was concluded that the most suitable material that can be used to treat sandy soils with gypsum (even for very high gypsum content) is the asphalt emulsion.

Chapter 8

Conclusions and Recommendations

8.1 Conclusions

From all the studies that have been presented in this dissertation, the following conclusions can be listed.

Conclusion 1: Sands with medium to high gypsum experience much lower volume change and mass losses during wetting-drying cycles when mixed with asphalt emulsion than activated fly ash.

Gypsum sands treated with asphalt emulsion or activated fly ash shrink, with a few exceptions, as the number of wetting-drying cycles increases. The degree of shrinkage is reduced as the dosage rate increases.

At the highest dosage rate of 30% activated fly ash, the lowest volume change at the end of the test was -8.8% (shrinkage) for medium gypsum sand cured for 7 days and +3.0% (expansion) for high gypsum sand cured for 28 days. However, neither meets the volume change target of ±2.5%.

At the highest dosage rate of 18% asphalt emulsion, the lowest volume change at the end of the test was -1.9% for medium gypsum sand and -2.8% for high gypsum sand. It should be noted that the lowest volume change of -2.2% for high gypsum sand was achieved at a dosage rate of 12% asphalt emulsion. These findings show that asphalt emulsion in dosage rates of 12-18% can control the volume change to within ±2.5%.

Activated fly ash was unable to control mass loss of sands with medium and high gypsum contents. Specimens with medium gypsum content suffered mass loss of at least 45% for dosage rate of 30%. None of the specimens with high gypsum content survived the complete set of 12 wetting-drying cycles, and thus a final mass loss could not be calculated.

At the highest dosage rate of 18% asphalt emulsion, the mass loss at the end of the test was 5% for medium gypsum sand and 4% for high gypsum sand. At this rate, the mass loss is less than the target of 7% and even lower than the reference specimens which has been treated with Portland cement.

Conclusion 2: The rate and amount of gypsum dissolution in sands with medium to high gypsum can be reduced when mixed with asphalt emulsion or activated fly ash.

For specimens treated with activated fly ash and survived all the 12 wetting-drying cycles, the lowest gypsum dissolution rate was approximately 1 g/L and the lowest cumulative dissolved gypsum was approximately 8 g for medium gypsum sand treated with 30% activated fly ash and cured for 7 days.

For high gypsum sand, the gypsum dissolution rate was approximately 1.1 g/L and the cumulative dissolved gypsum was approximately 7 g for specimen treated with 30% activated fly ash and cured for 28 days.

Medium gypsum sand treated with 18% asphalt emulsion showed the lowest gypsum dissolution rate of approximately 0.1% with an accumulative dissolved gypsum of 0.7 g, whereas, for high gypsum sand, the lowest gypsum dissolution rate of approximately 0.3% with an accumulative dissolved gypsum of 5 g was obtained by treating the soil with 18% asphalt emulsion.

As can be seen from these results, asphalt emulsion is more effective to decrease gypsum dissolution when compared to the activate fly ash. It is important to mention that the general gypsum dissolution rate is about 2.6 g/L at 25ºC and a pressure of 0.101 MPa.

Conclusion 3: A non-linear reduction in the permeable porosity was obtained when both soils treated with an increase dosage of asphalt emulsion.

The lowest permeable porosity in both soils was accomplished when they treated with 18% asphalt emulsion. In medium gypsum sand, the lowest permeable porosity was approximately 0.06 compared to approximately 0.19 for specimen treated with Portland cement. For high gypsum sand, the lowest permeable porosity was approximately 0.04 compared to 0.18 for specimen treated with Portland cement.

These findings are related to the cohesion and physical attraction between gypsum molecules and asphalt binders. Moreover, the coalescence process, which occurred after all the water of the emulsion evaporated during curing stage, helped to close the voids inside the specimen and that prevented more water form flowing through the specimen.

Conclusion 4: Untreated and asphalt emulsion treated samples showed a reduction in the soil coefficient of permeability (k) with the increase in the water volume, and the approach of mixing soil with asphalt emulsion, compacting, and curing achieved the lowest k and gypsum dissolution. In addition to that, the modified Baena and Toledo (2014) model provided a good prediction for the coefficient of permeability.

The coefficient of permeability (k) for untreated medium gypsum sand decreased from 8.5×10^{-3} to 1.9×10^{-3} cm/sec., with a total amount of dissolved gypsum of approximately 105 g, which represented approximately 48% of total amount of gypsum in the sample.

The value of k for untreated high gypsum sand decreased from 3.5x10^{-2} to 9.9x10^{-3} cm/sec., with a total amount of dissolved gypsum of approximately 490 g toward the end of the test, which represented approximately 79% of total amount of gypsum in the sample. The reduction in k values is related to the post gypsum dissolution compaction. Also, it should be noted that the duration of these tests was approximately one month, meaning that if these tests were allowed to be open for longer time, more gypsum would be dissolved.

On the other hand, the approach of mixing, compacting, and curing the 18% asphalt emulsion samples of both soils showed the lowest k values and gypsum dissolution. For treated medium gypsum sand, the value of k decreased from 1.3x10^{-3} to 1.2x10^{-5} cm/sec., with a total amount of dissolved gypsum of approximately 71 g, which represented approximately 51% of total amount of gypsum in the sample.

For the case of the treated high gypsum sand, the value of k decreased from 2.0x10^{-4} to 3.2x10^{-7} cm/sec., before the sample reached a state of no more water leached out of the sample, the total amount of dissolved gypsum was approximately 37 g, which represented as approximately 9% of total amount of gypsum in the sample. The treated samples test was done in flexible wall permeability cells, but with constant head procedure. Moreover, the duration of these tests was approximately 10 months, which is longer than the untreated samples test.

This approach of treatment showed that more gypsum can be prevented form dissolving by increase the dosage of the asphalt emulsion. Also, the process of compacting and then curing resulted in more adhering and physical attraction between asphalt binders with soil and gypsum particles.

From the trials of Baena and Toledo (2014) model on untreated soils samples, the results showed that the model did not match the actual testing results and provided a trend of an increase in the coefficient of permeability for both soils with the increase in the dissolved gypsum volume, which is the opposite of the tests results, as this model has not been designed for high percentage of soluble materials, and that what was found when this model was tested for variety of n_o and Ø.

Thus, a modification was done on this model and it has been used on the untreated and asphalt emulsion treated samples from both soils. The modified model showed a good match with all the coefficient of permeability results. Moreover, the modified Baena and Toledo (2014) model provided a good prediction for the coefficient of permeability in soil with gypsum, especially for lower values of dissolution (lower volume of soluble solids (Ø)).

Conclusion 5: Laminar water flow conditions increased the gypsum dissolution linearly when compared to static water conditions for both soils samples treated with 6% asphalt emulsion.

In the case of medium gypsum sand treated with 6% asphalt emulsion and went through cycles of wetting-drying in a container represented static water conditions, the gypsum dissolution average rate was approximately 0.16 g/L and the total percentage of dissolved gypsum was approximately 3.5%**.**

While the gypsum dissolution average rate was approximately 2.45 g/L and the total percentage of dissolved gypsum was approximately 20% for medium gypsum soil sample treated with 6% asphalt emulsion and subjected to laminar flow during constant head permeability test.

For high gypsum sand treated with 6% asphalt emulsion and went through cycles of wetting-drying in a container represented static water conditions, the gypsum dissolution average rate was approximately 0.37 g/L and the total percentage of dissolved gypsum was approximately 9.5%, compared to gypsum dissolution average rate of approximately 2.32 g/L and a total percentage of dissolved gypsum of approximately 78% for soil sample treated with 6% asphalt emulsion and subjected to laminar flow during constant head permeability test.

Conclusion 6: The unconfined compressive strength (UCS) increases with increase in the gypsum content with all treatments for specimens survived the all the wetting-drying cycles.

For activated fly ash treated specimens which survived all the wetting-drying cycles, the UCS for medium gypsum sand treated with a dosage of 30% and cured for 28 days was approximately 371 kPa, while for high gypsum sand treated and cured with the same conditions, the UCS was approximately 973 kPa.

The same behavior also was found for asphalt emulsion treated specimens which survived all the wetting-drying cycles, for 12% asphalt emulsion dosage, the UCS for treated medium gypsum sand was approximately 543 kPa, and 701 kPa for treated high gypsum sand.

Moreover, for 18% asphalt emulsion dosage, the UCS was approximately 971 kPa for treated medium gypsum sand, and 1353 kPa for treated high gypsum sand. Also, for the Portland cement reference specimens, which survived all the wetting-drying cycles, The UCS was approximately 2244 kPa and 5052 kPa for medium and high gypsum sands, respectively.

These results showed that the presence of gypsum in sand increased the strength for all treated specimens. For the case of activated fly ash, gypsum reacted with the activate fly ash by producing many geopolymer gels with the ettringite and that results in the increase in soil strength.

For the case of the asphalt emulsion, the presence of gypsum in the soil will increase the adhering and the physical attraction between the asphalt binders and the calcium sulfate molecules, which as a result will stiffen the structure of the soil.

On the other hand, Portland cement treatment will produce more CSH gel with more ettringite when the soil has more gypsum. However, this treatment is not desired for treating soil with gypsum due to self-sulfate attack during wetting.

8.2 Recommendations

Based on the results of this study, four major recommendations are proposed for future research.

1- Evaluate asphalt emulsion treatment on sandy soils with lower gypsum content.

It is recommended to evaluate the effects of asphalt emulsion treatment on sandy soils with gypsum contents as low as 5% and up to 25%. Previous studies from different regions around the world indicated that it is common to have soil conditions where gypsum does not dominate the soil composition.

Studies have also shown that damage can occur to the superstructure when constructed on soils with gypsum content as low as 5%. The initial recommendation is to use a wider range of dosage rates, up to 18%, of asphalt emulsion mixed with low gypsum sands.

There is evidence of a direct relationship between asphalt binder content and calcium sulfate content, as the most significant improvement in soil properties were achieved when sands with medium to high gypsum content were treated with 18% asphalt emulsion.

Using a range of asphalt emulsion dosage with soils that have lower gypsum content will help to establish a correlation between gypsum content and the optimum asphalt emulsion dosage.

2- Evaluate the effects of mixing asphalt emulsion or liquid asphalt and activated fly ash on the properties of gypsum sands.

The second recommendation is to explore the effectiveness of combining asphalt emulsion with activated fly ash. The use of 18% asphalt emulsion decreased permeable porosity and controlled volume change and mass loss to within tolerable limits.

The most significant gains in unconfined compressive strength were observed in specimens treated with 30% activated fly ash and cured for 7 days. Therefore, a mixture of 18% asphalt emulsion and 30% activated fly ash could improve strength and stability while reducing the potential for gypsum dissolution. However, the combined dosage rate might not be feasible, and as such, an appropriate amount and ratio of the two additives needs to be studied.

High dosage rates of asphalt emulsion are also not feasible due to the high proportion of water to asphalt in the emulsion. As observed in this study, mixtures with higher dosage rates of asphalt emulsion behaved more like slurry when mixed, making it difficult to prepare and compact the treated soil in a mold.

Adding activated fly ash might help to mitigate this problem because of the higher water demand of the ash. An alternative solution is to replace asphalt emulsion with liquid asphalt to eliminate the excess water.

3- Investigate the effects of NaCl concentration in water on gypsum dissolution in gypsum sands.

It is recommended to repeat experiments on 18% asphalt emulsion treated soil specimens with saline water to investigate changes in soil properties under wetting-drying conditions and continuous water flow.

This study showed that continuous water flow dissolved more gypsum when compared to static water conditions. However, these studies were limited to tap water that contains a low concentration of soluble salts, where the sodium chloride concentration is expected to be less than 75 mg/L.

Previous studies concluded that gypsum dissolution increases when other salts concentrations present in the water, a presence of sodium chloride (NaCl) increases gypsum dissolution three times the case of water without NaCl (Hardie 1967; and López et al., 1999, cited by Morillas et al., 2009). Therefore, its recommended to study the effects of different NaCl concentrations on gypsum sands properties.

4- Modify the collapse potential test to accommodate continuous water flow.

The standard collapse potential test does not allow for water to circulate through the soil sample inside the consolidation cell.

Therefore, a modification of the experimental setup and procedures is recommended to simulate the effects of continuous water flow in the field, in an effort to provide more representative measurement of collapse potential.

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